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Synthetic Sapphires.

By K. W. BROWN,
The British Jewel Manufacturing Co., Ltd. (G.E.C.)

R. C. CHIRNSIDE, F.R.I.C., L. A. DAUNCEY, B.Sc., and H. P. ROOKSBY, B.Sc., F.Inst.P.,
G.E.C. Research Laboratories.

INTRODUCTION.

THE word "synthetic" has all too frequently become synonymous in some minds with "artificial" and this, in its turn, with something which is merely an imitation of the genuine article. Synthetic sapphires are real sapphires, made by fusion of aluminium oxide and subsequent crystallisation. They differ from natural sapphires in a number of ways. With present techniques of manufacture they are strictly limited in size, whereas natural sapphires have been found weighing as much as 300 lbs.

They are, generally speaking, chemically purer, the colourless or "white" sapphire being composed of very pure aluminium oxide, $Al_2 O_3$. The synthetic ruby, another form of sapphire, will of course contain a considerable proportion of chromium oxide, but this cannot be thought of as an impurity. The colour of the blue sapphire may be produced by the use of such compounds as iron and titanium oxides, as additions to the aluminium oxide base.

The synthetic stones are generally free from flaws such as may be seen in natural stones and, ironically enough, are considered to be less valuable as gems on this account.

The original incentive to produce sapphires synthetically was for use in the jewel trade where values are related not only to optical quality, but also to the relative scarcity of the natural stone. Indeed, much effort has been expended in the past in attempting the preparation of diamond synthetically and in the main without any success. The recent discovery of Bannister and Lonsdale[1] that Hannay's diamonds made in 1880 are in fact true diamonds, is the first evidence of any success in this field.

Although sapphire, too, was originally made for its qualities as a gem, its other industrial uses have for long been the main object of the large scale industrial production.

CRYSTALLOGRAPHIC FEATURES.

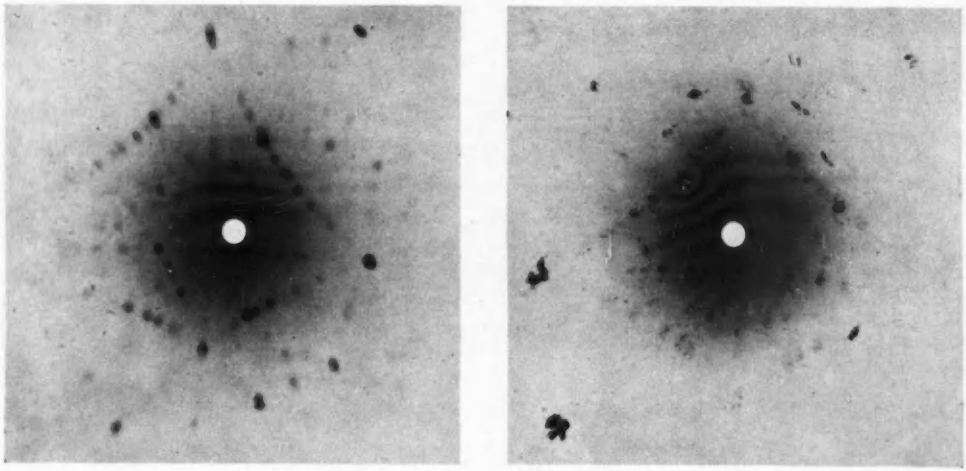
Sapphire, ruby, "oriental" emerald, corundum, are all forms of α-alumina and have fundamentally the same crystal structure. The term emerald is usually reserved for a stone similar in composition to the beryl. The true structure of α-alumina[2] is based upon a unit rhomb of apex angle 55° 17′ and side of length 5·42 A, but for many practical purposes the structure may be regarded as hexagonal with the side of the hexagon or a axis equal to 4·74 A and the height of the unit prism, the c or optic axis, equal to 6·45 A.

Sapphire is one of the hardest amongst minerals. On Moh's scale it is given the figure of 9, higher than quartz at 7 or spinel at 8. Although diamond, the hardest material known, is only 10 on this scale sapphire is much softer than diamond and not, as might be suggested from the figures, 9/10ths as hard. The scale is not numerically linear. Because of its hardness, powdered alumina has been utilised extensively as an abrasive or polishing medium. The toughness of the natural stones suggests immediately that instrument jewels made of sapphire will give particularly good wearing properties.

A crystal of pure alumina is normally without colour, but the alumina has the capacity of taking certain impurities into solid solution. For instance, ruby contains dissolved chromium oxide, and the solution is accompanied both by an increase in the dimensions of the alumina lattice and by the

remarkable change of colour from white to red. That the alumina crystal lattice is profoundly affected by the solution of the impurity is shown by the fact that ruby fluoresces strongly in ultra-violet light whilst "white" sapphire does not.

In blue sapphire the dissolved impurities are iron and titanium oxide, whilst in "oriental emerald" iron alone is the colouring medium.



(a) (b)

Fig. 1.—(a) and (b).—X-ray Laue back-reflection photographs of sapphire.

Natural sapphires differ in several ways amongst themselves and from the ideal single crystal of alumina. According to their origin they differ in depth of colour, and although large crystals are found these are relatively rare and the small imperfect crystal is more plentiful. Gem stones are selected both for perfection of colour and absence of gross visible flaws, but for industrial use other considerations may apply.

An interesting speculation about any form of natural crystal concerns how far it approaches the ideal single crystal condition in which continuity of structure extends to the external boundaries of the specimen. Many natural stones, although retaining an external shape characteristic of the particular crystal structure, are visibly broken up and fractured internally and, in many instances, may be mere agglomerates of fine grains intimately bound together in the form of the stone concerned. In other instances visual inspection does not reveal any discontinuities in structure and the stone is apparently without flaws. X-ray study does, however, reveal that finer differences exist and, in the case of natural sapphire, that the really perfect crystal appears to be a rarity.

X-ray Laue photographs may be used to demonstrate such imperfections in structure. The Laue photograph of a relatively perfect single crystal of sapphire is shown in fig. 1a and should be compared with that of a typical natural sapphire of imperfect structure shown in fig. 1b. The break-up and spread of the Laue reflection spots discloses that this sapphire is an intimate assembly of fine crystal fragments slightly disoriented with respect to one another, the angular disorientation amounting to, perhaps, a degree or more. The stone is mosaic in the sense that there are these departures from perfection of structure, but there are no gross flaws, and such stones can be cut and polished so that imperfections are not detected except by the X-ray method of inspection. That these features have an important influence on other physical properties of the stones is very probable, but the implications have not so far been thoroughly explored.

The synthetic sapphires grown in the Verneuil type of furnace described in a later section are closely similar to natural sapphires in that they are single crystals of α-alumina. The slow method of growth ensures that a relatively perfect continuity of structure is maintained, and certainly, except for accidents in

production, gross flaws are entirely eliminated.

In the early days of synthetic sapphire manufacture several misconceptions concerning the crystallographic features of the "boule"* arose. The longitudinal splitting of the boules into two parts was said to indicate a twinned structure, the separation taking place on the plane of twinning. The application of X-rays to investigate this point has shown that this is not so and that the plane of splitting is not even in general a principal crystallographic plane. The split appears to be entirely the outcome of the release of strain resulting from the temperature conditions existing in the boule furnace.

Another suggestion, that the crystallographic optic axis of sapphire is coincident with the axis of the boule, has also been shown to be incorrect. The only consistent crystallographic feature that has been found so far is that the optic axis in general lies in or very close to the plane of splitting; it may, however, make an angle anywhere between 0° and 90° with the axis of the boule. No explanation has as yet been offered to account for this coincidence of the crystallographic axis with the plane of splitting, but the determination of the direction of this axis may be important if sapphire jewels having the best wearing properties are to be cut.[3]

In general, the synthetic sapphire appears to approach the perfect single crystal condition more closely than the average natural stone. X-ray examination has been carried out on a large number of boules and no instance has been found of a good quality boule exhibiting such large imperfections of structure as are exemplified by the photograph, fig. 1b. This is not to say that imperfections in structure do not occur, but the kind of discontinuity or disorientation mentioned in connection with natural stones is most frequently found in boules which split badly or crack on cooling. Moreover, when an imperfect texture is detected it may not extend throughout the whole of the boule but may be confined to one end or to quite local regions. As with natural stones, it will be of interest to ascertain whether such departures from crystal perfection have any practical importance; it is not inconceivable that a physical property such as hardness should be influenced by the degree of departure from the ideal single crystalline condition.

* "Boule."—The usual term for the synthetic crystal, from the French for ball; Verneuil made small stones which were practically spherical.

HISTORICAL SUMMARY.

1. Early Work.

The early attempts to make artificial clear corundum varied considerably in detail but in general principle they fell into three broad classes. One method relied on the direct melting of alumina, another on treating compounds of aluminium so that they were decomposed to yield either solid or fused alumina, and in the third alumina was dissolved in suitable fused salts or fluxes and the whole kept hot for long periods so that the solvent evaporated and crystals of alumina were deposited. The oxy-hydrogen blowpipe, the foundation of the method by which success was finally achieved, was used to produce the high temperatures necessary in many of these investigations.

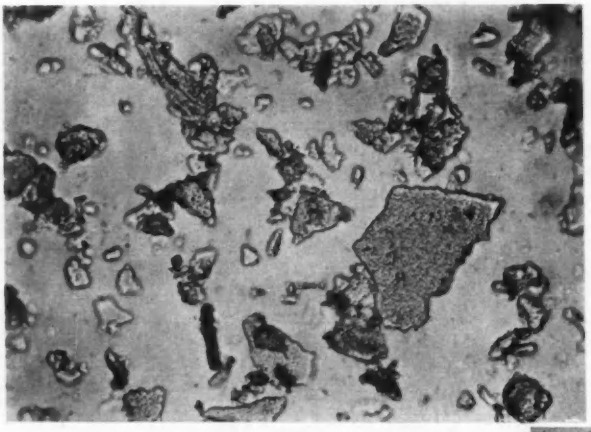
The first experiments made with the deliberate object of producing transparent corundum artificially appear to be those of A. Gaudin[4] who in 1837 obtained globules of fused alumina by strongly heating ammonium or potassium alum in carbon-lined crucibles. Although the globules were quite clear when molten they always became opaque on cooling. In spite of this, contemporary workers confirmed that they consisted of corundum and also found that if potassium chromate had been added to the alum small beads of ruby could be identified in the residue. Gaudin made many experiments in attempts to prevent the material from becoming opaque on cooling, probably gaining encouragement from the fact that he had no difficulty in producing a clear material by fusing quartz, but he had little success with the alumina.

In his last published notes in 1869[5] he virtually admitted defeat, commenting on the very great tendency of alumina to "devitrify." Other workers found the same difficulty and many came to the conclusion that alumina could not exist in the vitreous or glassy state and that their search was hopeless. Much work was probably abandoned because it was not then fully realised that clear natural corundum should not be regarded as vitreous like glass or fused quartz, but as the crystalline material it is now known to be.

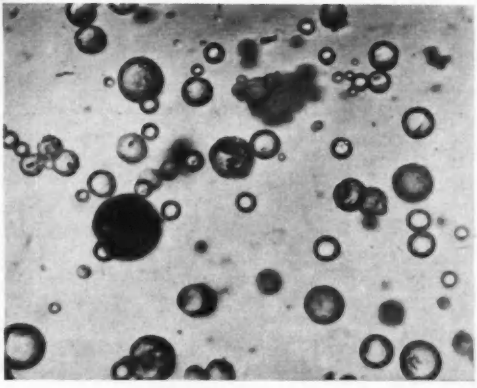
But this early work was not wasted, for it helped to lay the foundations of the industry which to-day produces many tons of fused alumina for use in abrasives, refractories, insulators and many other products. Deserving mention here is, perhaps, the contribution of Moissan, who in 1892 published particulars of the process of fusing alumina in an electric furnace.[6]

2. "Reconstructed" Gems.

There was one device which had a brief period of success at the end of the nineteenth century, although it cannot really be regarded as a true synthetic process. The products were known as "reconstructed gems" and were prepared by fusing together small chips of natural gemstone—usually ruby—or later by fusing small pieces on to one fair sized fragment to make a large stone. It does not appear to have been a very reliable process and it was not uncommon for such jewels to shatter, but until they were superseded by synthetic gems these stones found markets, and their quality must have been fairly good for a suspicion grew up that some were sent to India to be mixed with genuine natural stones and sold in that guise.



(a)



(b)

Fig. 2 (a) (above).—Photomicrograph (x275) of alumina powder used for making synthetic sapphires. (b) (right).—Photomicrograph (x275) of alumina powder after falling through the flame. The particles have become fused into spherical drops.

3. The Verneuil Process.

A workable process for making synthetic stones was eventually originated by M. A. Verneuil, who in 1904 published a description[7] and discussion of his method. This showed a very clear understanding of many of the difficulties and misapprehensions which had beset his predecessors.

In designing his apparatus Vernueil set out with three main considerations in mind: *a.* The alumina should be melted without overheating, for although the melting point of alumina is high (about 2050 degs. C.), the boiling point is only about 100 degs. C. higher. If the mass was allowed to boil it might become opaque and full of bubbles on cooling. *b.* To grow the mass of alumina slowly by depositing successive thin layers. This would ensure thorough refining of the alumina by allowing bubbles resulting from gaps between solid particles to escape readily. *c.* To arrange the process of fusion so that the area of contact between the growing mass and the supporting material should be a minimum. The importance of this can be readily appreciated now that it is known that the piece of corundum produced by Verneuil's method is actually a single crystal. The opacity of the product obtained by earlier workers while often, no doubt, accentuated by the presence of small bubbles was mainly due to the fact that the mass was polycrystalline. If a mass of liquid alumina contained in a crucible, for example, is allowed to cool, crystallisation will begin at the walls of the crucible, as these will cool first, and will proceed from many different points resulting eventually in a network of polycrystalline material. Verneuil's success lay in avoiding the use of a crucible for the fusion.

His method was simple in principle and has since been altered only in detail. A description of the process as it is now carried out will serve to illustrate his invention, and this is given in a later section.

INDUSTRIAL PRODUCTION.

Before the present war the production of

synthetic gems had grown to very large proportions, principally near the location of the watch-making industry, for millions of jewels are used annually in the manufacture of watches. The chief centres of production were Monthey and Locarno in Switzerland, Bitterfeld and Zwickau in Germany, and Annecy and Garrie in France.

Some of these factories have several hundred furnaces and the total daily capacity of these plants according to Kraus and Slawson[8] is between 750,000 and 1,000,000 carats, that is, nearly 200 kilograms or roughly 4 hundred-weights a day. This includes a proportion of jewels for decorative purposes, some of which are not corundum but spinel, a rather softer stone prepared by adding magnesia (magnesium oxide) to the alumina. There is little doubt that these three countries supplied the great bulk of the synthetic gems used in the whole world, exporting a large proportion in the form of finished or partly finished jewels.

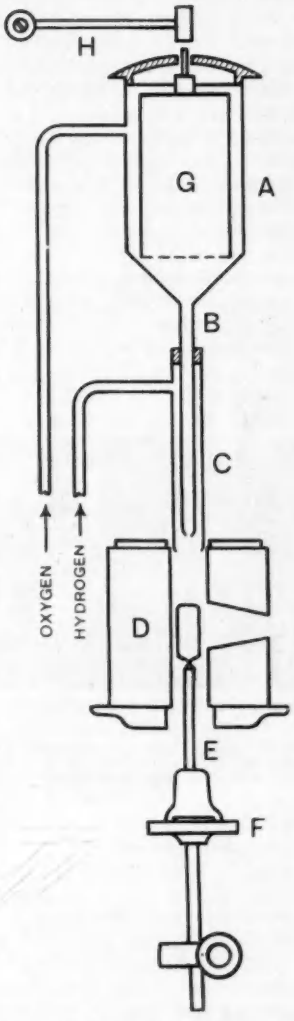


Fig. 3.—Diagram of Verneuil boule furnace.

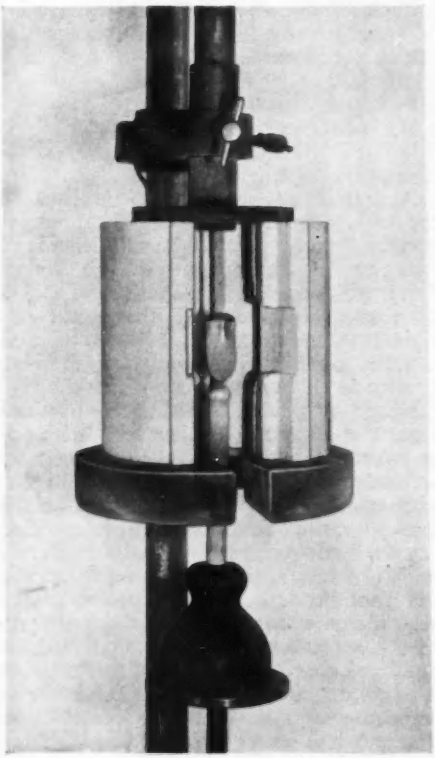
DEVELOPMENT OF THE BRITISH INDUSTRY.

When war became imminent it was realised that navigation instruments, chronometers, altimeters, compasses and the like, on which the safety of aircraft, warships, tanks and crews depended, were in their turn dependent on the possession of frictionless bearings, *i.e.*, sapphires and rubies. No British source of jewel bearings was available and, at the request of the Ministry of Aircraft Production, representatives were sent to Switzerland to study the process in operation and to make arrangements for setting-up a similar plant in this country. The British Jewel Manufacturing Co., Ltd., was formed to undertake this work. The original intention was to import the raw material from Switzerland and to install furnaces in this country for making the actual sapphires or "boules." Before this plan came to maturity, France had fallen and Switzerland was effectively cut off from direct contact with this country. Plans had therefore to include the preparation of the raw material and the development of the subsequent processes without further recourse to Swiss experience. In 1940 work commenced and sapphires were first produced from a limited stock of Swiss aluminium oxide. At the same time steps were taken in conjunction with the Research Laboratories of The General Electric Co., Ltd., to put into being processes for the preparation of suitable aluminium oxide and furnace bricks to withstand the severe temperature conditions, while provision for adequate supplies of hydrogen and oxygen and the replacement of furnace parts generally had also to be made.

The aluminium oxide is prepared by the ignition of ammonia alum of suitable purity. Fig. 2a shows a photomicrograph of a typical powder. The process of sapphire manufacture, in principle differing little from that originally conceived by Verneuil, consists of feeding this powder slowly through an oxy-hydrogen flame and causing it to fuse and subsequently to solidify and build up in a stalagmite-like way on a refractory support

or platform. By controlling the speed at which the powder is fed to the furnace, and therefore the rate at which the sapphire material builds up, a single crystal of sapphire may be produced.

A hopper, *A*, fig. 3, into which oxygen is fed by a pipe is extended downwards by a vertical tube *B*, ending in a jet. Surrounding this tube is a larger one *C* into which hydrogen is fed, so that tubes *B* and *C* together form an oxy-hydrogen blowpipe. The flame is directed



(a)

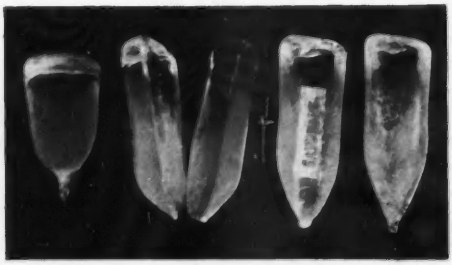
Fig. 4 (a).—A typical sapphire boule after cooling in the furnace.

downwards into the cylindrical central hole through the refractory brick *D* which, for convenience in handling, is split vertically into two similar sections. Projecting into the furnace so formed is a refractory "candle" *E*, supported on a table *F*; this can be raised or lowered by turning a handwheel. Fine alumina powder is placed in the suspended canister *G* closed at its lower end by a wire gauze cap. The burner is lighted and the small hammer *H* started so that it is lifted and allowed to strike the peg beneath at 2 or 3 second intervals. The canister *G*, is suspended from a flexible diaphragm, and at each blow of the hammer a small amount of powder falls through the gauze and is carried down in the oxygen stream. The alumina is melted in passing through the flame, fig. 2b, and is caught on the top of the candle *E*, which is in a cooler part of the furnace. Here a cone of sintered material builds up and as the temperature rises its apex just melts and forms a tiny spherical globule which grows upwards as more powder falls. As the globule grows in height the lower part is screened from the flame and solidifies; the sphere becomes a stem whose top may be made to spread by supplying more heat until it resembles a mushroom.

After a time the increasing heat supply is checked and the boule grows upwards without further increase in diameter. Finally the powder feed is stopped, the gas supply is cut off and the boule is allowed to cool in the furnace (fig. 4a).

A convenient size of boule is about 50–60 mm. in length, 20–25 mm. in diameter and weighs about 300 carats (1 carat = 0·2 gram.); this requires about 4 to 5 hours to grow.

After removing the cone of fritted powder a



(b)

Fig. 4. (b). — Typical boules, showing "splitting." The internal clarity can be seen from the half boule which is lying with its frosted outer surface on a label.

sharp blow on the stem will usually split the boule into roughly symmetrical halves the flat faces of which are essentially plane (fig. 4b). These half boules are in a convenient form for the subsequent operation of cutting into jewels. The general shape of the boule is a right cylinder, but occasionally a tendency to a hexagonal cross-section is noticed.

Pure alumina powder is generally used, giving a "white" sapphire for instrument

jewels and other industrial purposes, but coloured stones, rubies and blue sapphires, may be made by incorporating the appropriate substances with the alumina.

CONCLUSION.

An attempt has been made to give a picture of the development of the synthetic sapphire industry in this country. Although the Swiss industry has been established for many years, as has been explained earlier in this article it has not been possible to make full use of Swiss experience. Such progress as has been made in the relatively short time since work commenced has been a result of co-ordination of the experience so gained with the experimental work carried out at the Research Laboratories.

These British-made sapphires are now considered to be equal in every respect to the best that the Swiss manufacturers had to offer, but this is not to say that the process is incapable of improvement. Manufacturers have up to now worked along the lines originally laid down by Verneuil and the apparatus used is only a refined version of that originally designed by him. It is not inconceivable that some advantage, both in quality of sapphire and in the economics of the manufacture, might be achieved by substantial modifications in the process, and the research side of the question has not been ignored.

It is known that work has recently been carried out in the U.S.A., and advertisements have appeared in the technical press offering sapphires for industrial purposes, instrument jewels, tips for tools and the like.

In the U.S.S.R. experimental work commenced in 1928 in a small way, while in 1935 an extensive investigation was initiated by the Laboratory of Experimental Mineralogy of the Institute of Economic Mineralogy. Such information as is available suggests that progress has not been rapid and that difficulty has been experienced in making boules as large as those described in an earlier section.

A film was shown in London recently in which a demonstration of the Russian sapphire manufacture appeared. It seemed that the technique used was essentially that of Verneuil so that whatever developments may take place in the future it is clear that the method of setting about the problem as originally conceived by him was technically sound.

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Fig. 5.—Part of the furnace installation at a G.E.C. Works.

Quality Control when Manufacturing to Specification.

STATISTICS AS AN AID TO PRODUCTION EFFICIENCY.

By B. P. DUDDING, M.B.E., Ph.D., and W. J. JENNETT, B.Sc.(Eng.), A.M.I.E.E.,
G.E.C. Research Laboratories.

This article deals briefly with a subject treated more fully in "Quality Control when Manufacturing to Specification,"[1] recently published by The General Electric Co., Ltd. It describes methods which have been evolved to assist engineers in applying to problems arising in manufacturing industries the elementary statistical principles which have become generally known as "Quality Control."

FOUR articles have appeared in earlier volumes of this Journal dealing with various applications of statistical principles to industrial problems.[2,3,4,5] Since the last of them appeared in the issue of August, 1942, the writers have given single lectures or a series of lectures* to groups of engineers in various parts of the country. These lectures have in many cases been followed up by visits to factories to give assistance to those obtaining their first experience of the application of statistical methods.

The work has been associated principally, although not wholly, with the manufacture of articles to dimensional tolerances and the technique evolved is described in "Quality Control Technique when Manufacturing to Specification," published by the G.E.C.

GENERAL PRINCIPLES.

The following general principles have been found to assist production and inspection staffs to appreciate the technical position created by the existence of fixed specification tolerances.

First, if a considerable bulk of product is measured, the results can be described by two general characteristics: average level and variability. (See fig. 1 below).

Secondly, rejections by specification limits arise from errors in average level and/or too great variability acting independently or simultaneously.

Thirdly, the variability inherent in a process is that exhibited in the product manufactured in a relatively short time, say that exhibited by small samples of 2 to 6 specimens manufactured simultaneously or concurrently. (In processes it is usually that also associated with one batch of "raw materials"). It is the

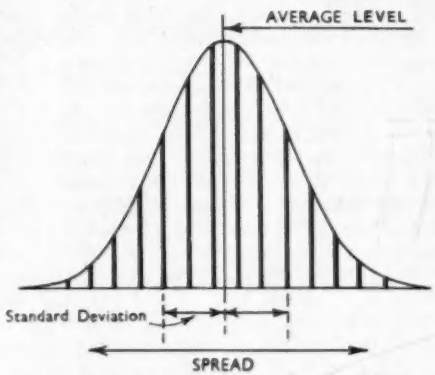

Fig. 1.—The distribution of measurements of a specified quality in a bulk product.

* Lectures have been given on 110 occasions to a total of about 4,000 engineers and personal discussions held with engineers on 80 occasions in organisations outside the G.E.C. In 1943 Dr. Dudding was appointed Hon. Lecturer in Industrial Statistics in the Faculty of Engineering, Sheffield University.

minimum degree of variability that can be expected in the bulk product, and can only be reduced by fundamental improvement in the machine or process.

Fourthly, an estimate of the process level at any time is given by the average of the measured values.

When production conditions are stable, in the sense that both the inherent variability and the process level remain sensibly constant for considerable periods, then the average values for samples conform to the control chart limits determined from the values of the process level and process variability. (See B.S. 600R—1942).

In practice it is generally found difficult to maintain processes strictly in the state of statistical control defined in the foregoing paragraph, and satisfactory production is not readily maintained unless the process variability is small relative to the specified tolerance.

To assist responsible engineers in appraising the manufacturing position the authors have introduced the concept of process precision relative to the specified tolerance. This is based on the third of the general principles already referred to :—*i.e.*, on the variability inherent in the process. This variability is conveniently measured by the *average* value of the range (difference between largest and smallest measured value in the sample) found to exist in the results for small samples containing the same number of articles, selected as indicated above.

CLASSIFICATION OF PROCESSES.

If process variability, measured by the average range in small samples, is small enough relative to the total tolerance permitted by the specification then *satisfactory* manufacture can be achieved without the results of measurements on samples conforming strictly to the control chart limits placed symmetrically either side of the manufacturing objective by amounts determined from the process variability.

Contrarily, if the process variability is large relative to the specification tolerance then the manufacture of rejects (product outside the specification limits) is inevitable.

There is an intermediate state when a satisfactory product can be made but the process level and the results for samples examined during patrol inspection must conform to the strict statistical limits obtained as described in B.S. 600R—1942.

Fig. 2 illustrates the three classes of manufacture where the distribution of measured values for individual articles is represented by a distribution of the normal form.

The magnitude of the total specification tolerance relative to the process variability is conveniently expressed by the ratio of the tolerance to the average range for sample of the same size. Table 1 divides manufacturing processes into the three classes referred to above, by using the value of this ratio, which the authors have called the *Relative Precision Index* (R.P.I.).

DISCUSSION.

In *low relative precision* cases an attempt should be made to increase the R.P.I. either by reducing the inherent variability by technical attention to the process or by increasing the specified tolerance. If neither is readily

TABLE 1.

CLASSIFICATION OF PROCESS VARIABILITY RELATIVE TO SPECIFICATION TOLERANCE.

Relative precision index (R.P.I.). = specn. tolerance/av. range.

Note.—This table should *not* be used if the range is out of control.

Class	Low Relative Precision	Medium Relative Precision	High Relative Precision
Sample size n.	R.P.I.	R.P.I.	R.P.I.
2	Less than 6.0	6.0 to 7.0	Greater than 7.0
3	„ „ 4.0	4.0 „ 5.0	„ „ 5.0
4	„ „ 3.0	3.0 „ 4.0	„ „ 4.0
5 & 6	„ „ 2.5	2.5 „ 3.5	„ „ 3.5
State of production	Unsatisfactory. Rejections inevitable.	Satisfactory, if averages are within control limits	Satisfactory, if averages are within modified control limits

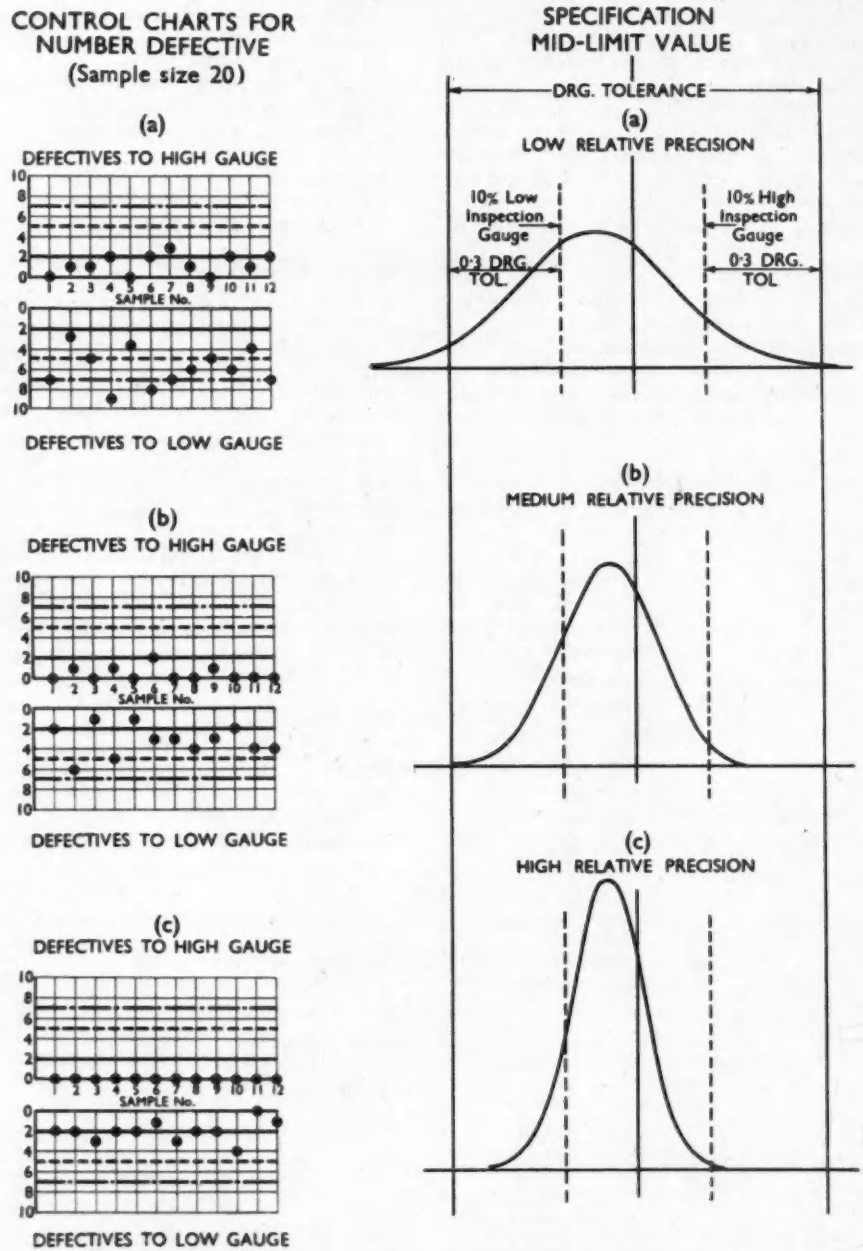

Fig. 2.—Illustration of the effect of variations in the relative precision of a manufacturing process on the control charts showing the numbers of articles failing to pass inspection gauges.

adjusted the process level should be maintained as near the objective as possible and watched by plotting the results of sample averages against the usual statistically determined limits.* Rejects will thereby be maintained at a minimum although not avoided. If the product is finally to contain no unsatisfactory articles it will require sorting by 100 per cent inspection.

precision permits, to give increased assurance against *production* of "rejects." This additional assurance may be desired because of the functional character of the product and/or because the tolerances are small compared with the commercially attainable repetition accuracy of measurement (that is small differences of opinion may arise between producer and consumer), and/or because sufficient

TABLE 2.

MODIFIED CONTROL CHART LIMITS FOR SAMPLE AVERAGE ($\bar{x}$) High Precision Class.

To obtain the limits, multiply $\bar{w}$ by the approximate values of $A''_{0.025}$ and $A''_{0.001}$, then add to the lower drawing limit and subtract from the upper drawing limit.

No. in sample n.	For Inner modified limits. $A''_{0.025}$	For Outer modified limits. $A''_{0.001}$	For alternative modified limits. $A''_{0.025}$	$A''_{0.001}$
2	1.51	0.80	2.32	1.60
3	1.16	0.77	1.70	1.30
4	1.025	0.75	1.46	1.19
5	0.95	0.735	1.34	1.12
6	0.905	0.72	1.26	1.08

Medium relative precision cases are usually best handled in much the same manner as low precision cases, unless the value of the R.P.I. lies towards the limiting values given for high precision cases. In such cases "rejects" can generally be avoided, although 100 per cent inspection of doubtful batches may have to be occasionally used.

When the manufacturing process belongs to the *high precision class*, satisfactory production is usually readily maintained. In order to avoid unnecessary adjustment to machines and/or processes the control chart limits can be modified by the use of Table 2. Instead of determining the control chart limits by "setting out" from the objective value by amounts determined from the process variability, they are "set in" from the specification tolerances, the amount by which they are "set in" being determined as usual from the process variability.

To obtain the limits, multiply $\bar{w}$ by the appropriate values of $A''_{0\cdot025}$ and $A''_{0\cdot001}$, then add to the lower drawing limit and subtract from the upper drawing limit. When the inner and outer limits are close together, one of them can be omitted.

In the above Table, alternative modified limits are also given for use, when the process information is not available to determine how nearly the distribution of individual values in the satisfactory product conforms to the normal form.

Fig. 3 illustrates how the modified control chart limits provide increased latitude to the process level when the relative precision of the process is high enough.

PROPORTION OF PRODUCT DEFECTIVE WHEN TESTED TO FIXED LIMITS.

The foregoing principles can be utilised even when it is either inconvenient or impossible to make measurements regularly during production. The authors have regularised a well-established works practice of using limit gauges for guiding shop practice which are more stringent than the usual final inspection gauges. This is achieved in the patrol inspection of the product during manufacture by using limits or gauges of such values that 10 per cent of the samples would on the average be classified as "defective" (not "rejects") when a just satisfactory product is examined. (By "just satisfactory" is meant a bulk product, the extreme individuals of which would just conform to the specified limit value). By this means the number of "defectives" to the inspection gauge found in small samples becomes appreciable before "rejects" to specification are manufactured.

If an estimate of the process variability is

* For modified procedures which are sometimes economically desirable, see Appendix E to "Quality Control Technique when Manufacturing to Specification," (see page 64).

available either from general knowledge, or as a result of measurement of a few small samples undertaken for this specific purpose, then the value of these inspection limits or gauges can be determined.

If no information whatever is available concerning process variability, then preliminary data to guide future practice can be obtained by using as 10 per cent patrol inspection limits or gauges, values set in from the specification limits by a proportion of the total specification tolerance. Whichever procedure is adopted the number of "defectives" found in samples selected from the production line is plotted against the control chart limits. Such a chart is illustrated in fig. 2.

The appearance of the chart provides an indication of the relative precision of the process and in some cases a 50 per cent gauge can be used with considerable advantage.

In the handbook to which reference is made a number of examples are discussed in detail, including the extension of the technique to cases presenting abnormal features.

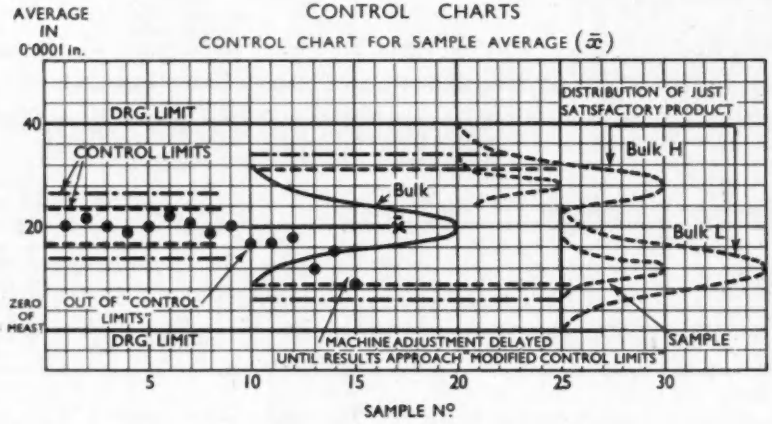

Fig. 3.—Chart illustrating the desirable manufacturing condition when process variability is small relative to specification limits.

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Fluorescent Tubes from the Viewpoint of the Maintenance Engineer.

By W. A. R. STOYLE, B.Sc., A.M.I.E.E.
(Osram Technical Department.)

INTRODUCTORY.

MOST maintenance engineers are quite familiar with the characteristics of tungsten filament lamps and of ordinary high pressure mercury vapour lamps and with the servicing of such lighting installations, but not many have very much knowledge of the fluorescent tube and its associated auxiliary equipment, as is only natural in what is a comparatively new field of the lighting art.

Such knowledge is becoming more and more desirable with the rapidly expanding use of fluorescent lighting, an expansion which undoubtedly will accelerate greatly in the post-war era. At present fluorescent tube lighting is necessarily restricted to essential and vital industrial and scientific work, but one can foresee the vast fields of industrial, commercial and even domestic installations which will inevitably demand this new and improved form of illumination.

The servicing of a fluorescent tube installation is materially helped by a thorough knowledge of the operation and construction of the tube itself and of its associated control gear. It is therefore proposed to consider in some detail those characteristics of the fluorescent tube and gear which will be chiefly involved in maintenance problems.

1. THE FLUORESCENT TUBE.

Although everyone is now quite familiar with the production of light by the passage of an electric current through a tungsten wire resulting in incandescence, the mechanism of light production by electric discharges through gases is not so fully understood. A complete understanding of this mechanism would involve a considerable knowledge of atomic physics, but the following explanation will suffice for the particular purpose in view.

Primarily, the electric discharge lamp consists of an envelope containing gas, such as mercury or sodium vapour or neon, into the ends of which are sealed two electrodes. When a voltage is applied across these electrodes a state of excitation is set up in the gas, in which collisions between the particles of gas take place. These collisions result in the liberation of energy in the form of light.

Unlike a heated metal filament which emits a continuous spectrum, that is all colours from violet to red, the radiation from electric discharge lamps is discontinuous, that is, only certain wavelengths (or colours) are emitted, each separate gas giving different colours or combinations thereof.

Not all the energy emitted by electric discharges is in the form of visible light, and in the earlier forms of discharge lamps much of this invisible ultra-violet energy was completely lost, as regards light production, being absorbed in the glass bulb of the lamp. More recently, however, use has been made of fluorescent powders which, when activated by ultra-violet light, themselves emit visible radiation. It is this principle which is used in the tubular fluorescent lamp, the tube being internally coated with special fluorescent powders. Certain powders, under the influence of ultra-violet radiation, fluoresce red, others blue and green and so on, and by combining these powders in a homogeneous mixture, practically any resultant combination of colours may be obtained. The major constituents of the fluorescent powder used in the present fluorescent tube, which give a more or less "daylight" rendering, are zinc, beryllium silicate and magnesium tungstate.

The discharge is activated at a very low pressure of mercury, actually about ·006mm., it being a characteristic of mercury vapour that at low pressures the passage of an electric current through it produces a greater proportion of ultra-violet radiation than at high

pressures. This ultra-violet light is of such a wavelength that it activates the specially chosen combination of fluorescent powders with the maximum efficiency.

Actually, only about 15 per cent of the light from the discharge itself reaches the eye as visible light, the other 85 per cent coming from the fluorescent coating which might be termed a "secondary radiator."

Sealed into the ends of the tube (fig. 1) are two electrodes which consist of coiled tungsten filaments coated with an electron-emissive material such as barium or strontium oxide. These electrodes are, of course, on alternating current, alternately anode and cathode. Both ends of each electrode are brought out to the contacts of a bayonet cap so that the electrodes may be pre-heated for starting purposes. A small quantity of argon gas is also introduced into the tube during manufacture, which has the effect of reducing the striking voltage and facilitates starting. The electric discharge through the argon gas has the effect of warming and vaporizing some of the mercury, enabling the discharge through the mercury vapour to commence. There is no appreciable light from the argon gas once the mercury has taken over.

As the electrodes on alternating current are alternately positive and negative, current must alternately flow into and out of the electrode coating. When the current is flowing out of the electrodes this is facilitated by the emissive coating, but in the reverse direction, that is, when the electrode is acting as an anode, the flow of current from gas to anode is more difficult and a voltage drop in the immediate vicinity of the electrode results. Two small metal fins are therefore connected to each electrode which facilitates the passage of current into the electrode, thereby helping to reduce the voltage drop.

In its present form the fluorescent tube is manufactured in one length only, 60 inches, and the diameter 1½ inches. It is rated at 80 watts and is made in two voltage ranges, 200/250 volts and 230/250 volts.

2. OPERATION OF THE TUBE.

At ordinary mains voltages a mercury discharge lamp of the type described would not strike unless steps were taken to reduce the initial "cold" resistance of the tube. Striking is therefore facilitated in two ways: first, by causing partial ionisation of the gas round the electrodes, and secondly by the creation of a high voltage surge in the circuit.

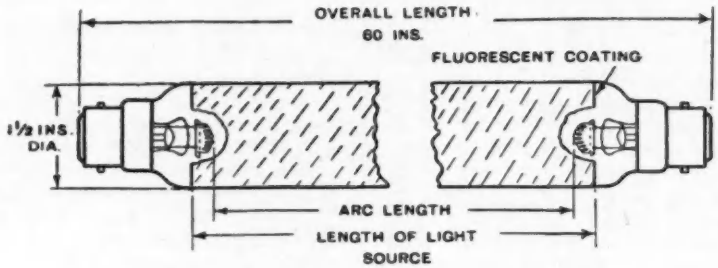


Fig. 1. Constructional details of 5 ft. 80 watt fluorescent tube.

When the lamp is first switched on, a device known as the "glow starting switch," which will be described in detail later, completes the circuit through the lamp electrodes, heating them for a few seconds at a current of approximately 1·3 amperes. Ionisation of the gas surrounding the electrodes having thus been instituted, the glow switch breaks the circuit and the collapse of the inductive field in the choke coil results in a momentary high voltage surge of about 700/800 volts across the electrodes, which is sufficient to initiate the discharge. During normal operation the current through the discharge is sufficient to heat the electrodes without the necessity for independent excitation.

The normal running current of the tube is approximately 0·8 amperes and the actual voltage across the tube, generally referred to as the "arc voltage," is about 115 volts. This latter figure is made up of a potential drop of about 15 volts in the immediate vicinity of the electrode and a uniform drop of about 100 volts along the discharge column.

3. CHARACTERISTICS OF THE TUBE.

(i) *Life.* Apart from failures due to accidental occurrences, such as glass cracks,

etc., the ultimate failure of the tube is caused by exhaustion of the electron emissive material on the filamentary electrodes, which is disintegrated slowly throughout life. A momentary loss occurs each time the tube is switched on, thus the number of switchings to which a tube is subjected has a certain influence on its life.

The rated average life of the fluorescent tube is about double that of a standard filament lamp and should be attained with the normal amount of switching which electric lamps undergo in practice.

Throughout the life of the tubes, the gradual disintegration of the electrode coating causes slight blackening at the end of the tube extending for a distance of two or three inches. This blackening is progressive and is a rough guide as to the period for which the tube has been in service. If, however, the lamp has been operating under abnormal conditions, such as excessive current, the tube will blacken prematurely which will afford some clue if such a fault is being investigated.

As with tungsten filament lamps, a certain number of lamps may fail before the normal rated life, due to inevitable small variations in manufacture. The percentage of such failures is, however, generally small, and abnormally long lives may frequently be achieved. For economic reasons, however, it is inadvisable to burn lamps much beyond their rated life, due to the diminution of light output.

When the electrode coating is exhausted, or near exhaustion, the mains voltage is insufficient to maintain the discharge and the starting switch continues to open and close, causing the lamp to blink on and off, a reliable indication that the tube requires replacing. It is most important that such a tube should be switched off quickly as the resultant current surges are likely to damage the glow starting switch and auxiliary gear. A further indication of a de-activated electrode is the deep orange coloured glow of the electrode, and very rapid blackening of the end of the tube.

Occasionally, dark streaks or patches appear along the length of the tube during life. This is due to condensation of the mercury vapour on the lower and cooler side of the tube. Rotation of the tube through 180° will give a more favourable position for evaporation.

In large installations it is sometimes convenient to operate some form of "group replacement" system, whereby batches of adjacent tubes which have had their normal life are removed, to avoid the necessity of having to replace odd tubes here and there in an installation at some awkward time when the maintenance staff may not be available.

(ii) *Light Output.* The light output of the fluorescent tube falls off gradually during life, partly due to the deposition of a film of sputtered electrode material on the fluorescent coating and partly due to actual chemical attack on the fluorescent coating by the mercury vapour.

The rated initial efficiency of the tube is 35 lumens per watt, that is, the initial light output at 80 watts is 2800 lumens. The light output falls off more rapidly during the first 100 hours or so, but after this initial drop the falling off is extremely gradual and at the end of the normal rated life the light output is at least double that of a metal filament lamp of equivalent wattage.

The fall in light output is not generally accompanied by any very noticeable change in the colour of the light, although some old

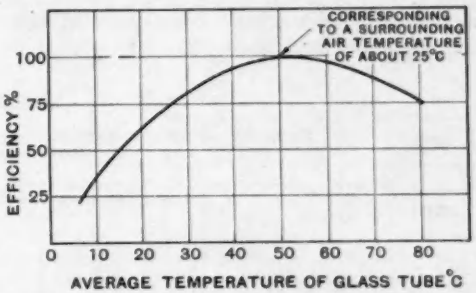


Fig. 2. The relation between luminous efficiency and the glass temperature of an unenclosed tube.

tubes may have a very slightly pink appearance. For this reason, also, and because of the depreciation in light output, bulk replacement is to be favoured rather than replacement of odd tubes here and there in an installation.

It is appropriate to stress here the desirability of keeping tubes and reflecting surfaces reasonably clear and free from dirt, particularly in view of the relatively large areas of tube and reflector involved.

(iii) *Effect of Surrounding Air Temperatures.* The luminous efficiency and the light output are largely determined by the powerful ultraviolet radiation at one particular wavelength, actually at 2537 Angstrom Units. The radiation at this wavelength is influenced by the pressure of the mercury vapour, which in turn is influenced by the temperature of the atmosphere in which the tube is operated.

The tube has its normal rated efficiency when burning unenclosed in a surrounding air temperature of 20°C. (68°F.) corresponding to an actual tube wall temperature of about 50°C. (122°F.) Fig. 2 gives a curve taken on a typical lamp, which shows the relation between luminous efficiency and the actual glass tube temperature of an unenclosed lamp. It will be noted that the luminous efficiency rises to 100 per cent of the optimum with increasing temperature up to an air temperature of about 25°C., and then gradually falls at higher temperatures.

It will be clear that at low temperatures, enclosed fittings tending to keep the temperature up to the optimum value, and also the avoidance of cold draughts, will be an advantage. Low temperatures may also make starting difficult, resulting in repeated flashing of the lamp before starting, with consequent deterioration of the electrodes and possible damage to the control gear.

(iv) *Stroboscopic Effect.* All discharge lamps, when burned in the usual manner on alternating current, have a non-uniform light output, as the light drops almost to zero with each half-cycle, thus causing a period flicker. This flicker is, of course, more noticeable at lower frequencies. However, the persistance of glow of the fluorescent powder helps to reduce this effect which, in most installations, is hardly noticeable.

A further form of flicker can be observed at the end of the tube, caused by the cathode glow forming on the electrode once each cycle, giving a flicker of half the frequency of the normal flicker. This flicker is usually obscured by covering up the ends of the tube.

Another flicker effect may occasionally occur in certain tubes in which the discharge takes the form of a narrow cord which spirals and oscillates against the sides of the tube. This is thought to be due to traces of carbon gases, but the effect is rarely noticed once the tubes are initially aged.

In installations where rotating machinery is employed, stroboscopic flicker can give rise to peculiar optical illusions such as to make the machine gear wheels appear stationary when they are actually rotating or to give rise to the multiplication of images. In such cases it is advisable to operate tubes on different phases of the supply and to arrange that there is a certain "overlap" of the light from different tubes so that the illumination on the machine never quite falls to zero at any one instant. Alternatively, the tubes may be operated in the special "twin tube" circuit which is described in a later section of this article.

Towards the end of life a 25 cycle flicker may be noticed which is due to unequal dissipation of the electrode coating and a consequent partial rectification effect.

(v) *Power Factor.* To consider the tube itself, the current is to all intents and purposes in phase with the applied voltage so that the power factor is near unity and the tube acts in much the same manner as a pure resistance. Actually, the power factor of the tube alone is about 0·9 because of the distortion of the wave form, this distortion being caused by the fact that the current through the discharge during each half cycle does not commence until the voltage has risen to a certain value (the striking voltage) and ceases to flow before the voltage falls to zero (the extinguishing voltage). However, the necessary use of an inductance in series with the discharge introduces a lagging power factor into the circuit, the power factor of the lamp-choke circuit being about 0·5. By using a suitable condenser of about 7½ microfarads capacity, the power factor of the circuit can be raised to 0·9, and by the use of the twin tube circuit unity factor can be achieved.

Table 1 above gives the capacities of condensers required to achieve different power factors.

TABLE 1.

Capacity of Power Factor Correction Condensers for Osram 80W. Fluorescent Tubes.

Mains Volts	Capacity in microfarads to give the following power factors					
	0.7	0.75	0.8	0.85	0.9	0.95
200—220	4.5	5.0	6.0	7.0	8.0	9.5
230—250	4.5	5.0	6.0	7.0	7.5	9.0

(vi) *The effect of Mains Voltage Variation.* When a metal filament lamp is operated on a higher voltage than that for which it is designed, both the total light emitted and the wattage consumed increase. As the light emitted by the lamp, however, increases at a greater rate than the wattage, the efficiency in terms of lumens per watt also increases. In the case of the fluorescent tube, however, an increase in mains voltage results in an increase in total light and in wattage consumed, but as the wattage increases at a greater rate than the light output, the efficiency in terms of lumens per watt actually decreases. This decrease in efficiency is due to the fact that the higher current density (that is the amperes per square centimetre of tube cross-section) produces the short ultra-violet radiation at 2537 Angstrom Units less efficiently, which radiation, as already stated, is largely responsible for the luminous efficiency of the tube.

Fig. 3 shows the effect of variation of mains voltage on the tube current, voltage, light output and efficiency. It is interesting to note that the light "regulation" with varying mains voltage of the fluorescent tube is considerably better than that of the metal filament lamp. With a metal filament lamp a 1 per cent increase or decrease in the mains voltage results in about a 3½ per cent increase or decrease in the light output. In the case of the fluorescent tube, however, a 1 per cent increase or decrease in the mains voltage only results in approximately a 1 per cent increase or decrease in light output.

The general effect of under-voltage operation is decreased light output and uncertain starting. Considerable under-running will also reduce the life, since the reduced current through the tube is not then sufficient to maintain the electrodes at their correct operating temperature. The general effect of over-voltage working is, of course, the overloading of the tube and its auxiliary equipment.

From the above it will be clear that to ensure correct and satisfactory operation of the tube, the auxiliary equipment must be correctly adjusted to suit the particular mains voltage concerned. As mentioned previously, overloading will cause premature blackening at the ends of the tube and the choke coil will also overheat. Serious overloading may even cause breakdown of the choke coil insulation.

(vii) *Colour.* The colour of the light emitted by the 80 watt fluorescent tube approximates very closely to that of noon sunlight. Fig. 4 shows in graphical form the luminosity distribution throughout the visible spectrum of the fluorescent lamp compared with that of noon sunlight on a clear June day. The close agreement will be obvious. The heights of the rectangles enclosed by the full lines are proportional to the total amount of light in each wavelength band radiated by the tube.

The ultra-violet radiation emitted by the 80 watt Fluorescent Tube falls in the harmless range of wavelengths—4000 to 3000 Angstrom Units. Actually the amount of energy received by an object at a short distance from the tube is less than that received

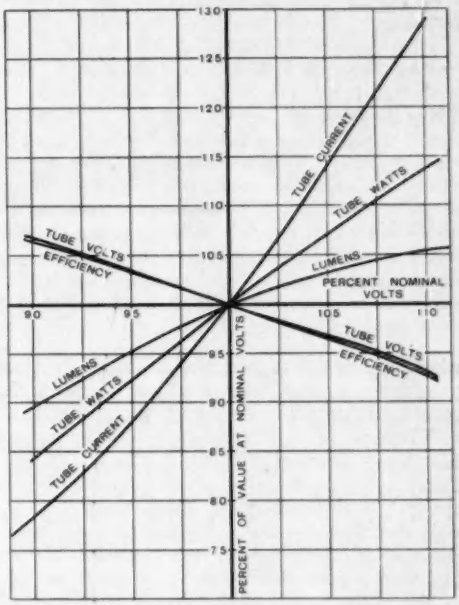


Fig. 3. The effect of variation of mains voltage on the characteristics of Osram fluorescent tubes.

even in winter sunlight and there is no radiation whatever in the danger zone, that is, below 2,800 Angstrom Units. This is due to the fact that the glass envelope of the lamp completely absorbs the very short wavelengths which are necessary to excite the fluorescent powders.

The relative amounts of ultra-violet energy at different wavelengths received from the sun in winter and summer compared with that received from an 80 watt Fluorescent Tube at a distance of five feet from the tube are shown in Table 2, which will make the above points quite clear.

(viii) *Radio Interference.* Electric discharges in general tend to generate high

frequency oscillations which may cause radio interference either by direct radiation from the tube and its associated wiring, or by "feed-back" through the supply leads. Generally speaking, this interference is only noticeable when the wireless receiver is in fairly close proximity to the tube itself, and when the receiver is tuned in to a very weak signal.

When wireless interference does occur the broadcast wavelengths 200 metres to 500 metres are generally most affected. In general, interference on short waves is not objectionable because the amount of interference at these short wavelengths is not high and is confined to relatively small sections of the short wavelength broadcast band.

For the above reasons, it is usual to shunt the tube by a small 0·05 microfarad capacity condenser, which is generally sufficient to suppress the radio interference. In some exceptional cases, particularly where the wireless receiver is very close to the tube, special precautions may be necessary.

In investigating the cause of radio interference, it is necessary first of all to ascertain whether the interference is due to feedback through the mains or to direct radiation. This can be ascertained by interposing an earthed wire mesh between tube and receiver. If the interference ceases, it is most likely caused by direct radiation from the tube, and one of the following adjustments should be made :—

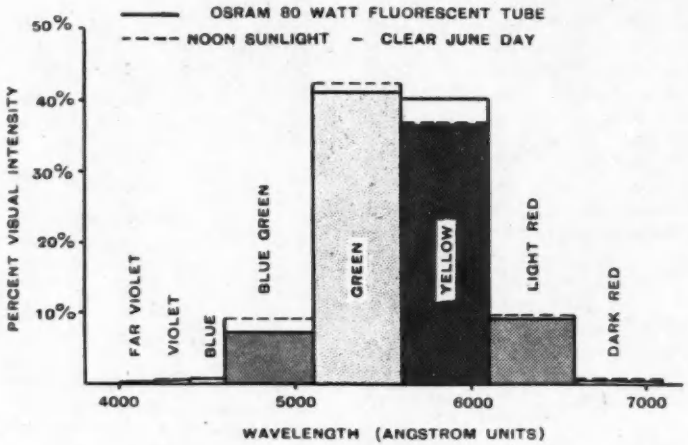


Fig. 4. Luminosity distribution of Osram fluorescent tube compared with that of noon sunlight.

TABLE 2.

Ultra-Violet Radiation from Osram Fluorescent Tubes.

Description of Radiation	Wavelength Region	Approximate values of Radiation in Milliwatts on 1 Sq. cm. of surface		
		Clear noon Sunlight in England		80W. 5ft. Fluorescent Tube at 5 feet
		Summer	Winter	
Near Ultra-Violet (harmless)	4000—3250Å	3.5	0.4	Less than 0.005
Erythemal Region	3250—2800Å	0.2	0.001	Less than 0.001
Harmful Short Wave-length Radiation	below 2800Å	None	None	None

(1) Reverse the lampholder at one end of the tube.
(2) If this fails to improve matters, reverse the lampholder at the other end.
(3) Try a counterpoise earth on the receiver.

On the other hand, if the interference is caused by direct feedback through the mains, a mains filter will probably be necessary, such as is illustrated in fig. 5. This filter should be connected as close to the fitting as possible.

The above remarks presuppose that the 0·05 μfd radio supression condenser normally connected across the tube is in working

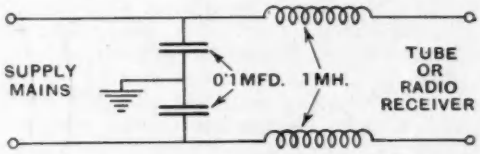


Fig. 5. A typical filter for suppressing interference radiated from the mains.

order. This might have developed a defect, and one from a satisfactory fitting should be substituted for it to check this point.

4. AUXILIARY GEAR.

Before discussing the actual fluorescent tube circuit, it will be helpful to consider the components of which it is constituted.

The essential components of the circuit are the lamp, series choke coil and glow starting switch. In addition to these items there are the radio suppression condenser (already described) and a power factor correction condenser. These condensers are, however, not essential for the operation of the tube.

(i) *Choke Coil.* For satisfactory operation of the fluorescent tube, the use of a suitably designed inductance or "choke" coil is necessary. Mercury vapour has a so-called "negative" resistance because, as the current through the tube increases, greater ionisation of the gas occurs which, in turn, results in a lower resistance, whereas with metal filaments increase in current through the wire results in an increase in resistance. Hence the choke is necessary to limit the current through the tube.

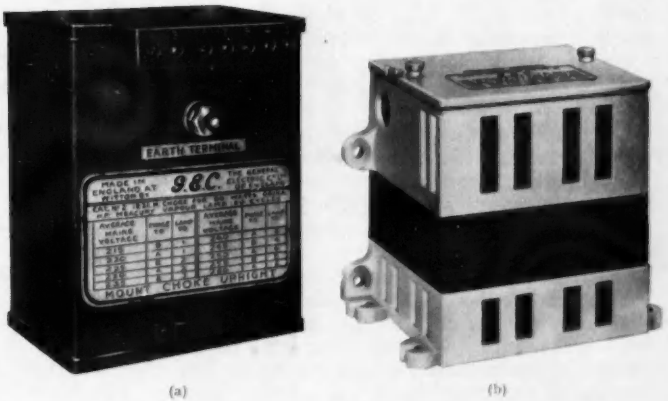
In addition to being a "current limiter," the choke must have several other important characteristics. Apart from limiting the normal operating current of the tube to approximately 0·8 amperes, the choke must pass a sufficiently high current through the electrodes of the tube for adequate pre-heating (about 1·3 amps.) before the tube has started. The choke must also be designed to ensure that the current in the circuit is not seriously affected by sudden fluctuations of supply voltage. In addition, the choke must have a low wattage loss, a satisfactory operating temperature and a low noise level.

80 watt chokes, embodying these characteristics, are made in two main types, the "open-protected" and the "solid," the wattage loss in each type being approximately 10 watts. Both types are illustrated in fig. 6 below.

The "solid" choke, in which the laminations and coils are embedded in wax, is rather more generally used because of its resistance to moisture, general robustness and complete freedom from any hum. Both types of choke are manufactured in two voltage ranges, 190 to 230 volts and 220 to 260 volts, and are tapped in 5 volt steps.

It is important that the chokes should be set at the "average" mains voltage so that the tube is operated at the correct nominal current. If there is any doubt as to just what the supply voltage really is, the necessary tests should be made before the installation is finally switched on, and the chokes set accordingly.

Faults in chokes are extremely rare, but the



(a) (b)

Fig. 6. Choke coils for use with fluorescent tubes : (a) solid type, (b) standard protected type.

failure of a choke can be brought about by some maladjustment or fault in the rest of the circuit. A short circuit in the radio-suppression condenser, or the contacts of the starting switch becoming stuck together, would cause an excessive current to flow through the choke and the tube electrodes, which in time would cause serious overheating of the choke coil. In such a case, the tube electrodes would remain continuously alight whilst the tube would make no effort to strike, which would clearly indicate the probable source of the fault. Apart from these considerations, a wiring fault in the fitting could short circuit the choke, overloading it, and disintegrate one or both of the tube electrodes.

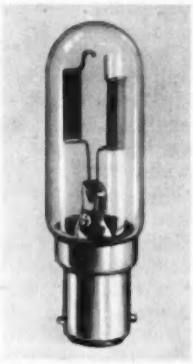


Fig. 7. Glow starting switch. Overall length, 70 mm., diameter of bulb, 20 mm.

(ii) *Glow Starting Switch.* The glow starting switch, which is in effect a "time delay" switch, is illustrated in fig. 7 above, and consists of two bi-metal strips which carry the switch contacts enclosed in a small tubular bulb which is filled with helium gas at a low pressure. A more recently developed form of this switch consists of a single U-shaped bi-metal strip engaging with a fixed contact.

As has been seen, the object of the glow switch is to provide a pre-heating current of 1·3 amperes through the tube electrodes for starting purposes, and as this current is in excess of the running current of about 0·8 amperes, it will be clear that the thermal delay characteristics will have a considerable bearing on the electrode pre-heating time and eventually on the life of the electrode itself.

For example, the over-quick breaking of the switch contacts will result in inadequate heating of the tube electrodes, causing prolonged flashing of the tube in an endeavour to start. Such a fault is unusual, but should it occur the particular fitting should be switched off before the tube is damaged, and the switch replaced.

It will also be clear that the striking and extinguishing voltage of the switch must be controlled by the manufacturer within close limits. If the striking voltage is too high the contacts will not close, and if the extinguishing voltage is too low the contacts may close after the tube has started, causing periodic flickering. A normal switch will have a life of many thousand switchings and will outlive several tubes.

5. CIRCUITS AND CIRCUIT OPERATION.

(i) *The Simple Fluorescent Tube Circuit.* Fundamentally, then, the essential components of the fluorescent tube circuit comprise the lamp, choke and starting switch. In addition, the circuit is usually supplied with a radio suppression condenser and a power factor correction condenser, all of which have now been described. The complete circuit is illustrated in fig. 8. It will be noted that a 100 ohm resistance is connected in series with the radio suppression condenser; this prevents the condenser discharging across the contacts of the glow starting switch and welding them together. The radio suppression condenser and 100 ohm. series resistance are made up in a single cylindrical unit with an internal fuse which opens the circuit if the condenser insulation breaks down.

It is interesting to consider in detail the actual mechanism of the starting circuit which can be divided into three successive stages, as follows :—

(*a*) The main switch S.1 is closed. This applies practically full mains voltage to

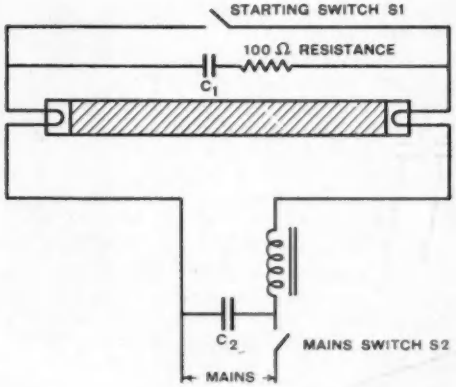


Fig. 8. Circuit diagram for Osram fluorescent tube.

the tube and to the glow starting switch, which is connected in parallel with the tube. The mains voltage is insufficient to start the main discharge in the tube, but the discharge in the helium gas of the glow switch is initiated and heats the bimetallic strips which bend over and make contact.

(*b*) The glow switch contacts having closed, the supply circuit is completed through the choke and the lamp electrodes back to the supply again. The current through the electrodes is limited to approximately 1·3 amps. by the choke coil. This current passing through the electrodes is sufficient to raise them to incandescence and to start ionisation of the gas in their immediate vicinity.

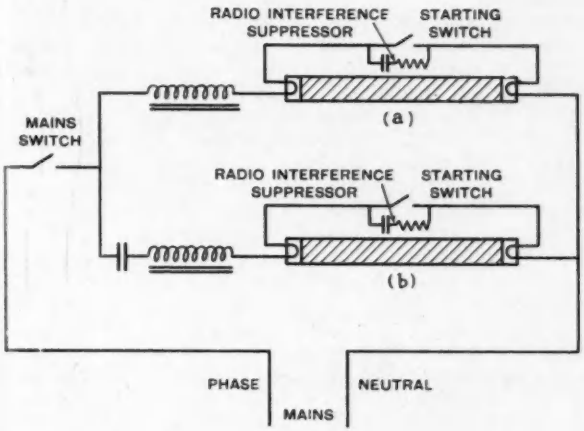


Fig. 9. Diagram of twin-tube circuit : (a) Inductively or choke controlled tube, (b) Capacitatively or condenser controlled tube.

(*c*) As the glow switch is now short circuited (the switch contacts being closed) there is no longer any heat from the helium discharge in the switch, and therefore the bimetallic strips cool, thereby opening the switch contacts in a second or two. This breaking of the circuit gives rise to a high voltage kick of about 700/800 volts caused by the collapse of the magnetic field in the choke coil, which is sufficient to start the discharge through the fluorescent tube.

After the tube discharge has started, the voltage across it is about 115 volts, the rest of the supply voltage being taken up by the choke. This voltage is insufficient to start the discharge in the glow switch so that the switch remains effectively out of operation until the tube has to be re-started again.

(ii) *Twin-Tube Circuit.* An interesting and recent development in fluorescent tube circuits is the twin-tube method of operation illustrated in fig. 9.

In this circuit one lamp is operated in the normal way in series with a choke coil and the other in series with a 6·25 μf capacity condenser and a choke coil. The circuit with the choke alone in series will have a power factor of approximately 60 per cent lagging, and that with the choke and series condenser a power factor of approximately 60 per cent leading. The resultant power factor of the twin-tube circuit is, therefore, practically unity.

As the light from each tube is in phase with the current in the tube, and the current in one tube is 120° out of phase with the current in the other, the light from the two tubes will also be 120° out of phase. Thus, at the instant of the cycle when one tube is completely extinguished, the other tube will still be alight, thereby evening out the resultant illumination on the working plane and considerably reducing the stroboscopic flicker. The improvement is about the same as that which would be obtained by operating two normal tube-choke circuits on two phases of a three phase supply.

MISCELLANEOUS FAULTS.

Apart from the various possible faults already mentioned, there are a number which cannot be classified under one or other of the above headings and which will now be considered.

The complete failure of a tube to show any light at all, apart from any actual discontinuity in the wiring or supply, must be due either to a broken lamp electrode or to the non-operation of the glow starting switch, both of which faults are easily traceable.

In regard to faulty wiring, this can, of course, cause immediate destruction of the tube electrodes, glow switch and eventually of the choke coil itself. If only one electrode of the tube is glowing, an earth in the starting switch circuit is indicated. An earth between choke and tube will short circuit the choke, allowing a catastrophic current to pass through the tube, whilst the choke may, by a wiring

fault, become short circuited across the mains and will overheat in a very short period.

Due to the fact that during the starting period momentary voltages of 800 to 1000 volts are produced, breakdown may occur in the wiring or in the radio-suppression condenser if the insulation of these components is not of good quality. Generally speaking, an inspection of the effect of the overload will lead one quickly to the cause.

It should be stressed that a circuit which is not behaving normally should be disconnected at once from the supply until the defect can be remedied. If a number of tubes are operating from the same circuit switch, the tube should be removed from the defective unit and this particular circuit locally isolated.

In conclusion, it will be evident from the foregoing that, whilst the maintenance of a fluorescent tube installation is not quite such a simple matter as that of a tungsten lamp installation, it is certainly not complex, and a study of this method of illumination will be more than repaid when the advantages in efficiency and colour to be gained by the use of fluorescent tube lighting are considered.

The Chemistry of Plastics.

By R. C. CHIRNSIDE, F.R.I.C.,

G.E.C. Research Laboratories.

The development of the plastics industry has been so rapid during recent years that it has seemed to be a matter of some importance to provide a concise and easily understandable review of the fundamental chemistry of the subject.

This article has been adapted from a lecture recently given by the author.

INTRODUCTION.

MUCH has been written about the subject of Plastics, and the layman is now conscious that there have been spectacular developments in the variety and in the uses of them during the last few years. It is doubtful, however, if exact knowledge concerning the nature of these materials is any more widespread than it is about dyes, for example. The public continues to refer to these as "aniline-dyes" regardless of their real composition or, indeed, of what aniline is or where it comes from. So, too, in some minds all plastics are "Bakelite," and this has become something of an embarrassment to the Bakelite Company. Most publications of a general type have been concerned with the properties and the uses of the various plastics; in the present article the more restricted field of the chemistry of these materials will be considered, and although this subject is in itself very wide it should be possible to get an intelligible picture of what has gone on, of what is going on and possibly, even, of what is coming, in this fascinating branch of chemical endeavour.

Some small amount of chemical knowledge will be taken for granted and chemical formulae will be used but it will soon be evident that these serve to simplify and not to complicate matters.

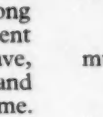
One of the hardest things to do is to define

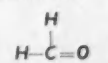
a Plastic, for many plastic materials do not come under this classification at all and few of the materials that do can be said to be plastic. Indeed, most Plastics are hard, rigid substances which have in the course of their fabrication gone through a plastic state. It should be noted that clay, rubber, putty are plastic but are not generally considered as Plastics. The word is usually associated with plastic organic materials from which rigid or semi-rigid objects may be made. The processes may embrace moulding, casting or extrusion, with or without the application of heat.

Now the term "organic material" has been used and it will be well straight away to see what this implies. Most people are now familiar with the conception that all material and living things in the universe are composed of permutations and combinations of some of the 92 elements, and that of these 92 only 50 or so are at all common. Organic chemistry was the term originally applied to the chemistry of those compounds which, it was erroneously believed, could not be prepared artificially in the laboratory but were formed solely in living organisms under the influence of a mysterious agency termed the "Vital Force." Faith in this vital force was seriously shaken, however, in 1828 when Wöhler showed that Urea, one of the most characteristic products of the animal metabolism, could be prepared from the inorganic constituents cyanic acid and ammonia (fig. 7). It is interesting to recall that at one time a serious attempt was made to classify chemical knowledge into animal, mineral and vegetable sections, along the lines of the old party game. The present distinctions of inorganic and organic have, however, served their purpose very well and are likely to persist for some time to come. Indeed, it is very difficult for a chemist to attain, and more difficult for him to retain, an expert knowledge in both branches. The most striking fact that should be noticed, for it is of the greatest importance in connection with the subject, is that the bewildering collection of organic chemicals or compounds is brought about by permutations and combinations, not of the 50 or so common elements previously mentioned, but usually of 3 and sometimes less frequently of 2 or 3 more.

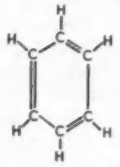
The Elements.

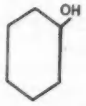
The 3 principal partners in this business are carbon, written as *C*, which is known in its more usual forms as coal, coke and soot and less frequently as diamond. The other two partners are both gases, hydrogen, *H*, and oxygen, *O*. In combination these two form water, H_2O, and singly are known to us by their uses and effects rather than by their appearance. Of the less prominent or sleeping partners, the main one is nitrogen, *N*, another gas, and sulphur and phosphorus sometimes take a small part in the proceedings. That part of the human frame other than the bones is wholly composed of these few elements. The only other chemical conceptions which need be mentioned are those of atoms and molecules. A molecule of any given compound is the smallest possible particle or unit into which the compound can be divided and yet retain its chemical properties unchanged. It is usually a group of atoms bound together by, what for simplicity may be called, chemical affinity. The modern physicist and physical chemist have ways of explaining chemical affinity that are outside the scope of this article and it will be convenient to stick to the simpler conceptions of linking forces or valency.

Everything in organic chemistry and, incidentally, in the synthesis of plastics turns on the fact that carbon is always quadrivalent, *i.e.*, has four bonds available for attaching itself to other atoms, like and unlike. We can write carbon thus: the four short lines may be thought of as arms. Oxygen has but two and may be written $-O-$ or $=O$, and hydrogen but one, $-H$. If we are to represent combinations of these atoms we must write them thus:  or CH_4 for example would represent the gas methane (miner's firedamp). Acetylene would be C_2H_2 or $H-C\equiv C-H$, and it will be noticed that the chemist satisfies his conscience, and the carbon atom, by writing in three bonds between the two carbon atoms. The compound is said to be "unsaturated" thus recognising that it still has a number of unrealised aspirations.

If we write  we have again kept faith with the carbon atom and have written down our paper conception of what formaldehyde should look like. Obviously the arrangement in space is not limited to the two dimensions of the paper but the convention will

serve. If we are to be more ambitious we shall presently come to that characteristic symbol of the organic chemist the hexagon. This is chemical shorthand and is usually written

but in its more extended form appears like this  , it is C_6H_6 or benzene. This time the quadrivalent nature of the carbon atom is shown by the alternate double linkages in the cyclic compound.

If we perform a small chemical operation on the benzene ring thus:  it becomes phenol, C_6H_5OH, popularly known as carbolic acid. These formulae are all set out in fig. 1.

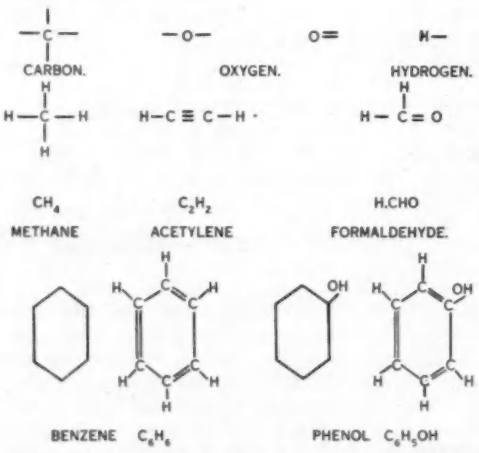


Fig. 1.—Formulae of some of the commoner starting materials.

"Bakelite."

In 1909, Leo Baekland, an American chemist of Belgian origin, carried out the synthesis of one of the most important plastics, for he took two of these compounds and was able to make them interact under appropriate conditions. In chemical language, he condensed phenol and formaldehyde to give a product which was resinous in character and which we know as Bakelite. This reaction is shown in its simplest form in fig. 2. By adjusting the conditions and the proportions of the reacting substances we can eventually

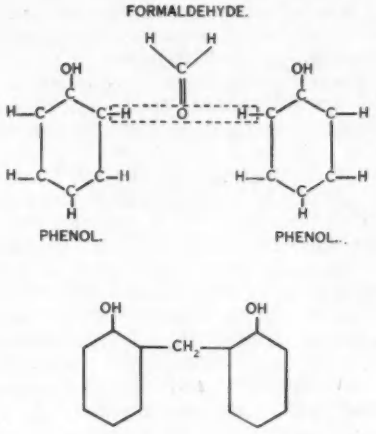


Fig. 2.—Molecule of phenol-formaldehyde.

arrive at a mixture composed of long chains of molecules of irregular constitution as shown in fig. 3. It has been calculated that if all the possible primary valency links were satisfied the tensile strength of a phenol-formaldehyde resin should be theoretically 2,500 tons per sq. inch, whereas the actual value is about 5 tons per sq. inch. The accepted explanation of this is that there are flaws in the structure of the network, possibly because the nuclei do not lie as conveniently in one plane as shown in fig. 3. If we represent the arrangement of the internal structure as in fig. 4 it is clear that in the positions marked 2 and 7, for example, the CH_2OH group and the OH group are not so likely to bridge across as they would be in the arrangement shown in the previous figure. It will already have been realised that besides the linking up of the phenol and formaldehyde to form a new compound the various groups of this compound have also begun to link up with one another, not only sideways but so as to form a veritable network of cross-linked molecules. This is illustrated diagrammatically in fig. 5.*

The various stages in the setting or hardening ("curing") of the bakelite type resin are

* Reproduced by permission, from "Plastics for the Engineer," A. Reyrolle & Co., Ltd.

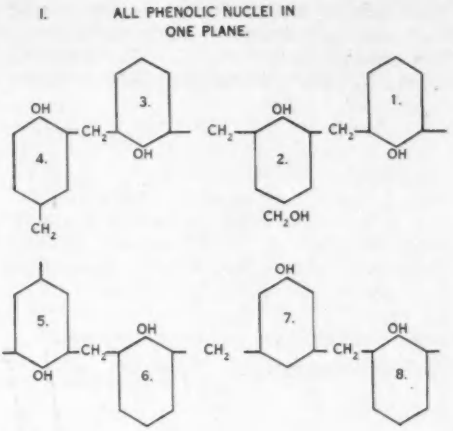


Fig. 3.—Macro-molecule of phenol-formaldehyde resin: all phenolic nuclei in one plane.

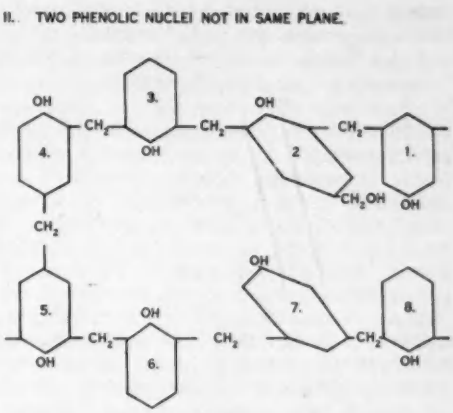


Fig. 4.—Macro-molecule of phenol-formaldehyde resin: two phenolic nuclei not in the same plane.

therefore related to the successive steps in the chemical combination leading eventually to this intricate network which goes to make, and is typical of, what we call a thermo-setting resin or plastic.

CLASSIFICATION.

Plastics are conveniently divided into two classes, thermo-setting and thermo-plastic. Those compounds which in their original condition will flow and can be moulded by heat and pressure, but which in their finished or "cured" state cannot be re-softened or moulded, are known as thermo-setting. Thermo-plastics, on the contrary, can be re-softened and re-moulded by heat.

THERMO-SETTING RESINS.

Phenol-Formaldehyde (P.F.) Resins.

The thermo-setting materials are usually supplied as a powder with an appropriate filler, if required, consisting of wood flour, slate dust, mica, asbestos or the like. The resin is in one of the earlier stages of combination and the application of heat and pressure brings about further combination until in the appropriate circumstances the fullest combination is brought about giving a hard infusible and insoluble moulding.

The simplest type of thermo-setting resin is the phenol-formaldehyde compound, but this is merely representative of a whole class of phenolic compounds. Phenol is the simplest member of the family, many members of which will react with formaldehyde in a

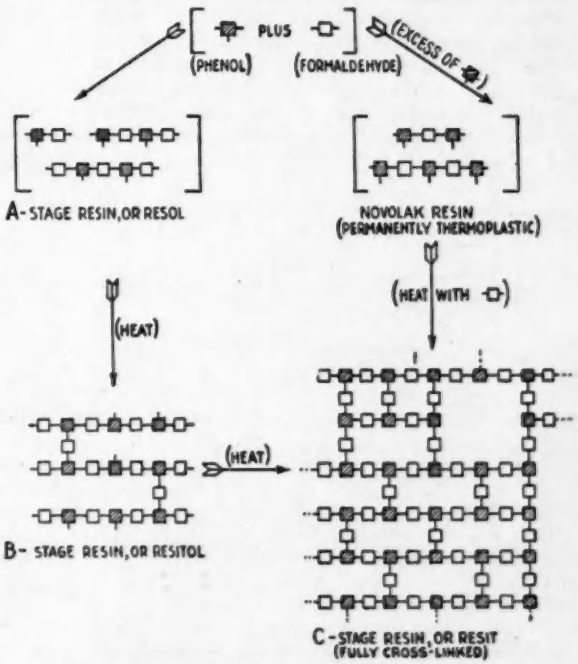


Fig. 5.—Formation of phenol-formaldehyde plastics.

similar manner. If we add a CH_3, or methyl group, to a phenol a cresol is obtained; if two CH_3 groups are added a xylenol is produced. Moreover, depending on the respective points of attachment of these CH_3 groups, any of 3 cresols, all having the same chemical formula but each having a different space arrangement of atoms and having different chemical properties, may be formed. This is illustrated in fig. 6, where the more complicated case of the xylenols is also shown, for we may have 6 of these according to the disposition of the CH_3 groups. The 3 phenolic compounds most likely to form resins are phenol, meta-cresol and symmetrical meta-xylenol, each of which has 3 positions that may be attacked during the first stage of resin formation. Different condensation products are obtained depending on the point of attack, and the commercial resins available do, of course, comprise some of these complex compounds. Although the resins produced are not unlike the natural resins such as colophony or pine rosin they are not related chemically. Besides their use in moulding powders they are used alone or after reaction with natural resins to form the basis of some modern paints. In this case one of the requirements is that the resin or resins used shall be oil-soluble. Other uses with which most people are now familiar are in the impregnation of paper, cloth, wood and as a glueing or bonding agent in some special circumstances. The P.F. type resins represent about 75 per cent of the world production of plastics.

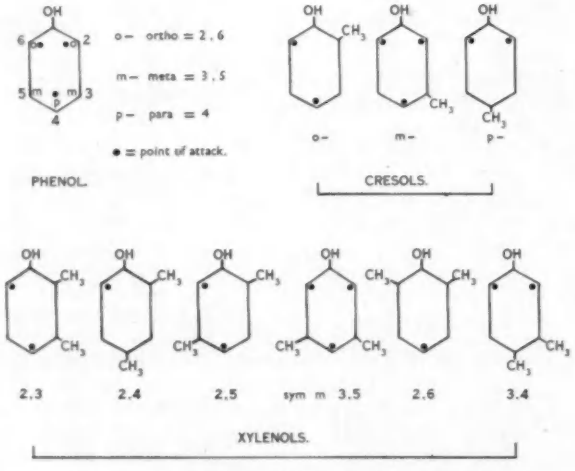


Fig. 6.—Structural formulae of phenolic compounds.

Urea-Formaldehyde (U.F.) Resins.

The other major thermo-setting plastic is that produced from urea and formaldehyde, best known perhaps under the trade name Beetle. This has uses and properties generally similar to the P.F. resins but has certain advantages, *e.g.*, the resins are colourless and can be tinted to delicate pastel shades. They also have better non-tracking properties for electrical applications.

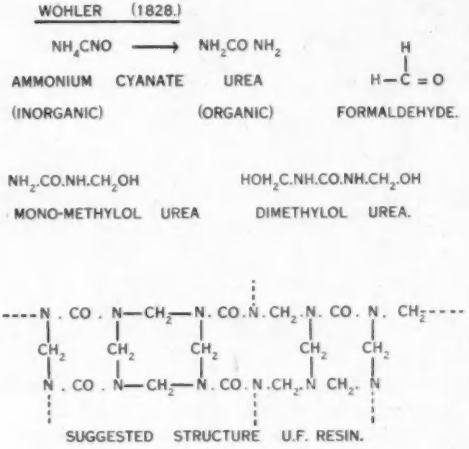


Fig. 7.—Suggested structure of urea-formaldehyde resin.

Urea, or carbamide, will undergo condensation with formaldehyde; the first stage of the reaction is usually carried out by dissolving urea, $NH_2.CO.NH_2$, in aqueous formaldehyde below 10°C. and the condensation is effected at 130°C. Water is removed and the molten resin is run off into trays to cool. The structure of the U.F. plastics is not definitely known; initially mono-methylol urea $NH_2.CO.NH.CH_2.OH$ and later di-methylol urea is formed, $CH_2.OH.NH.CO.NH.CH_2.OH$. Further condensation then takes place and some of the suggested reactions are shown in fig. 7. The important thing to notice is that the structure is again a cross-linked network as in the P.F. type.

Melamine and Aniline Resins.

Among the most important of the other thermo-setting resins are the melamine and aniline formaldehyde resins. The first promises to be a very important ingredient of paints and enamels, as indeed the urea plastics already are, while the aniline plastic has special non-tracking properties for electrical applications. "Panilax" is one of the commercial varieties of this type. In the U.S.A. there are available also soya-phenol-formaldehyde and phenol-furfural resins. The first of these is an offshoot of Ford's experiments in soya bean production, while the second is a by-product of the Quaker Oats Co., the furfural being obtained from oat hulls. A plastic "Cafelite," has also been produced from some part of the coffee bean.

Alkyd Resins.

The glyptals or alkyd resins are usually included in the thermo-setting class but their chief application is in the paint industry and they never harden or "cure" to a rigid or brittle product like those discussed earlier. They were developed originally in America and are formed by the reaction of polyhydric alcohols such as glycerin with dicarboxylic acids such as phthalic acid. A simple example of the reaction is shown in fig. 8; for the production of oil soluble resins suitable for paint manufacture, a combination of fatty acid, glycerol and dicarboxylic acid is brought about, and the resulting product constitutes the basis of the remarkable "synthetics" of the paint manufacturer which have already threatened to displace nitrocellulose lacquers in many fields.

THERMOPLASTICS.

We may now pass on to a consideration of the other main class, the thermoplastics, for it is here that the chemist has had his greatest and most recent triumphs, more particularly with polymers and macro-molecules.

Cellulose.

The earliest plastic is celluloid, essentially a di-nitrate of cellulose, made by treating cotton linters with nitric and sulphuric acids. The nitrogen content is about 11 per cent; with a higher nitrogen content (17 per cent) the product is gun-cotton. To produce celluloid the nitrated cellulose is compounded or plasticised with about 35 per cent of its weight of camphor. The great disadvantage of celluloid is its inflammability and it was at first thought that the introduction of cellulose acetate which is relatively non-inflammable would displace the nitrate. However, because of certain small differences in properties it has not done so except for non-flam film and one of the forms of artificial silk.

The cellulose acetate is also prepared from cotton linters, the short staple fibres remaining after all the spinnable cotton has been taken out. The structure of cellulose, the basis of so many naturally occurring materials such as wood, flax, jute, cotton, etc., is complex and it is in fact a natural long chain molecule. The unit or brick from which the cellulose edifice is built may best be considered as a molecule of the simple sugar, glucose. In fig. 9 the formula for glucose is shown in its usual form and also re-arranged in cyclic form. By joining a large number of units of anhydro-glucose end to end, by the elimination of water from the molecule, the cellulose molecule can be built up. Actually, it is a great deal easier to break down the cellulose molecule to give the units of glucose, so that the "popular science" idea of making sugar from sawdust is not so fanciful as it might at first appear. The cellulose plastics are derived from cellulose by replacing the *OH* groups numbered 1, 2 and 3 in the figure by various acid groups; in the case of the nitrate by NO_3, in the acetate by CH_3COO groups from nitric and acetic acids respectively. The di-acetate has the most useful properties and the finished material is plasticised and may be dyed and sliced like

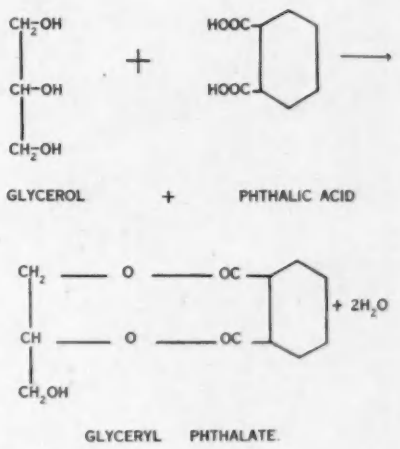


Fig. 8.—Molecule of "Glyptal" resin.

celluloid. For textile use the acetate is squirted through a die to give threads which may subsequently be spun. It is interesting to note that Sir Joseph Swan, the inventor of the electric lamp, in his search for a lamp filament, came near to producing the first synthetic textile. Indeed, some of the finer cellulose threads, produced by him from blotting paper and from which his carbon filament was subsequently formed, were crocheted by Lady Swan into lace for small mats and doyleys and a few of these articles were exhibited at the Inventions Exhibition of 1885 under the description "artificial silk." Swan was not unaware of the implications of his discovery but his hands were already full and he left the task of developing this material to others. Among the pioneers who worked out a practical process of making artificial silk from squirted cellulose was Charles Stearn, the bank clerk from Birkenhead who had been one of Swan's collaborators in the development of the electric lamp.

Swan's material, and what we now call viscose and the proprietary material "cellophane" are essentially pure cellulose, regenerated cellulose. That is to say, some form of naturally occurring cellulose-rich material like wood or paper is dissolved in certain chemicals and is reprecipitated from solution in a form which is purer than the starting material and is also in a convenient shape, say as a textile thread. Several new modifications of the cellulose plastics have been developed during the last few years, such as the mixed ester cellulose aceto-butyrate and also methyl, ethyl and benzyl cellulose. These all have claims to special properties in one direction or another.

STRUCTURE.

It is clear that these natural materials like cellulose are structurally long chains of molecules, the unit molecule or group being repeated at intervals along the chain. The application of X-ray analysis to cellulose has shown that it is built up of at least 100 glucose units joined end to end, and it is not unreasonable to connect the thread-like structure with the plastic properties of these materials. X-ray examination of the thermo-setting compounds on the other hand gives a complicated picture of the molecule, and although it is possible to make some limited identification of the different plastics (see fig. 10) it is fairly clear that the cross network arrangement of these molecules is very different from that of

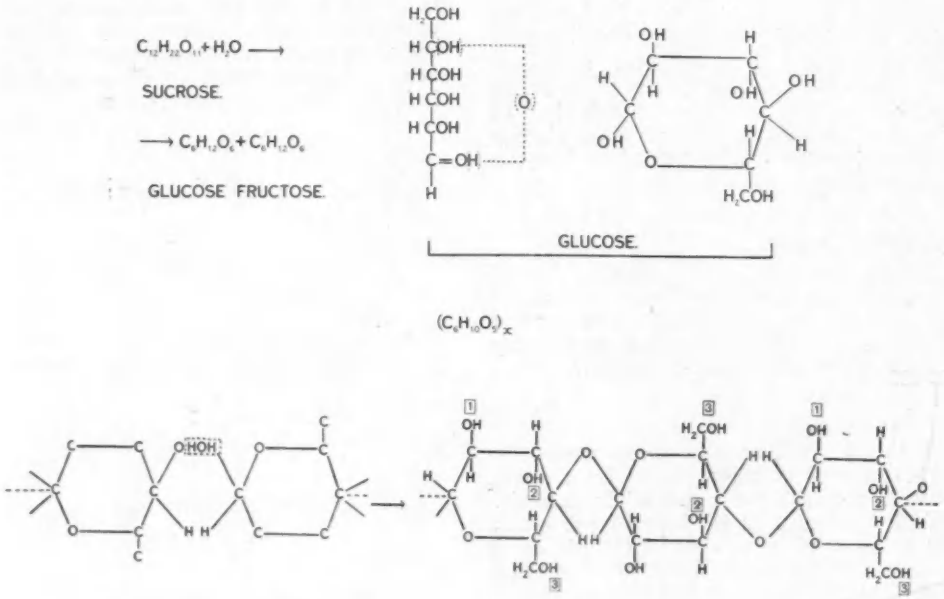
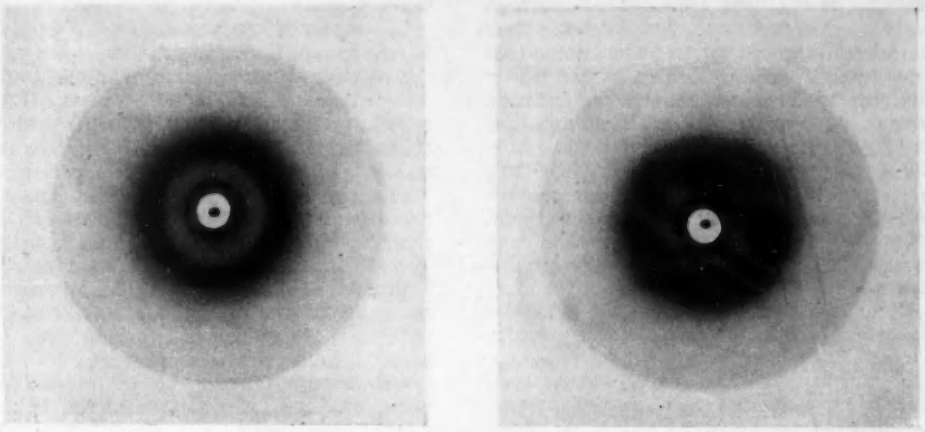


Fig. 9.—Linking of a number of glucose units to form a cellulose chain.



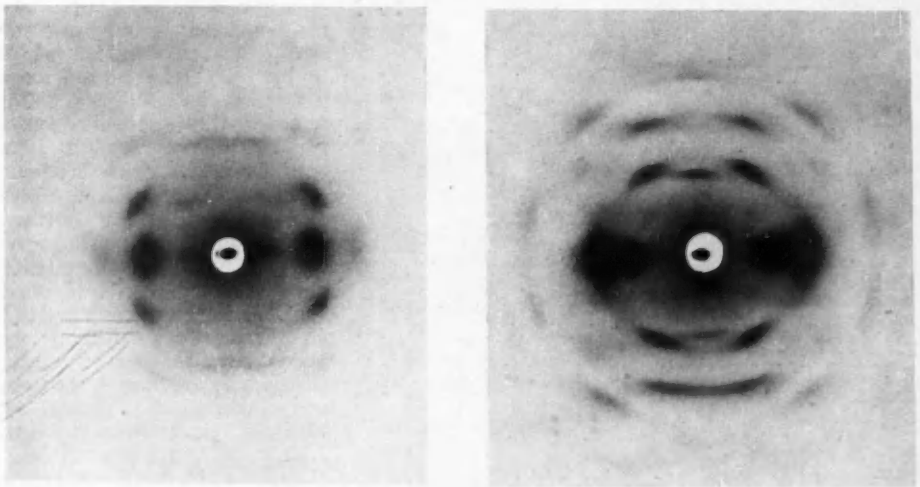
(a) (b)

Fig. 10.—X-ray diffraction patterns (Cu Kα radiation, 3.5 cms. distance) of: (a) phenol formaldehyde resin, (b) cresol formaldehyde resin.

the long chain compounds and does not permit of the same freedom of movement within the compound. In the natural long chained compounds the molecules are grouped parallel to one another; in materials like muscle, for example, they may be loosely linked together and a great deal of the strength and elasticity of natural fibrous materials is derived from this orientation of chain molecules into parallel bundles. But the geometry of the chains may differ; while the chain molecules of silk are nearly fully extended those of wool are zig-zag and crumpled which explains why wool can be so readily stretched.

Fig. 11 shows X-ray diffraction photographs of two natural fibres, silk (protein) and flax (cellulose). The X-ray patterns show that "crystallinity" is highly developed; in a high proportion of these natural fibres the molecules are regularly oriented parallel to the fibre axis so that crystalline regions are formed.

The chemist has tried to emulate nature and although he has not succeeded in duplicating synthetically the natural long chain



(a) (b)

Fig. 11.—X-ray diffraction patterns of natural fibres: (a) natural silk, (b) flax (cellulose) partially mercerised.

molecules he has been able to produce many new long chain products. Starting with small molecules, the monomer, the new compounds or polymers have been formed by linking up the units by a process known in chemical language as polymerisation. Celluloid and cellulose acetate about which we have already spoken are not really of this class for they are natural polymers to which the chemist has effected only minor alterations or additions.

HIGH POLYMERS.

Ethylene.

The simplest of these polymers is polyethylene (fig. 12), known also as "polythene" or "alkathene." This is a British product, developed and manufactured by Imperial Chemical Industries. Ethylene, $CH_2{=}CH_2$, is a gas which, under pressure and by the use of a catalyst, can be polymerised to a long chain compound having a molecular weight of the order of 30,000. The polymer is a white, tough but flexible waxy solid. It is thermoplastic and alone or plasticised with polyisobutylene, also shown in fig. 12, can be moulded by injection or compression, or by casting or extrusion and is available in the form of tube, rod, film, etc. It is chemically inert and has a very low power factor; indeed all its electrical properties are good and it is of special interest to the electrical industry.

Styrene.

If, in the ethylene molecule, a benzene or phenyl group, C_6H_5, is substituted for one of the hydrogen atoms thus: $C_6H_5.CH{=}CH_2$, a substance called styrene or vinyl benzene is produced (fig. 12). This is a viscous colourless liquid and it may readily be polymerised to form a glass-clear resin, polystyrene. The British commercial product made by the Distillers' Company is known as "Distrene." Polystyrene can comprise a chain of over 1,000 styrene molecules, and polymers have been made having a molecular weight of the order of one million. It softens at 70°—90°C. and is suitable for injection moulding. It has a high refractive index and good light transmission and may be used for optical purposes. It is, of course, available in many forms: sheet, tube, rod, etc. The electrical properties are outstanding and this material too is being widely used in the electrical industry.

Vinyl Plastics.

It will be noted that the term vinyl was used to denote the hypothetical grouping $CH_2{=}CH-$; this is ethylene with one H atom removed and has, of course, no real existence just as in inorganic chemistry NH_4, the ammonium radical, is used conventionally for something that has no real identity. However, a number of interesting and useful compounds are based on the vinyl grouping, for example, vinyl acetate and vinyl chloride can both be polymerised to give linear polymers as shown in fig. 13, and molecular weights of up to 80,000 have been recorded. The chloride, $CH_2{=}CH.Cl$, on polymerisation gives a substance that has been invaluable as a rubber substitute since the loss of our resources in the Far East. Polyvinyl chloride, or P.V.C. as it is usually called, can be obtained in a variety of forms and its resistance to chemical action, its low water absorption, its toughness and flexibility combined with good electrical properties have led to its extensive use for cable sheathing. It may also be obtained as a textile fibre, "Vinyon." The monomer is theoretically derived from ethylene and chlorine, $C_2H_4 + Cl_2$ but in practice acetylene and hydrochloric acid, $C_2H_2 + HCl$ are the starting

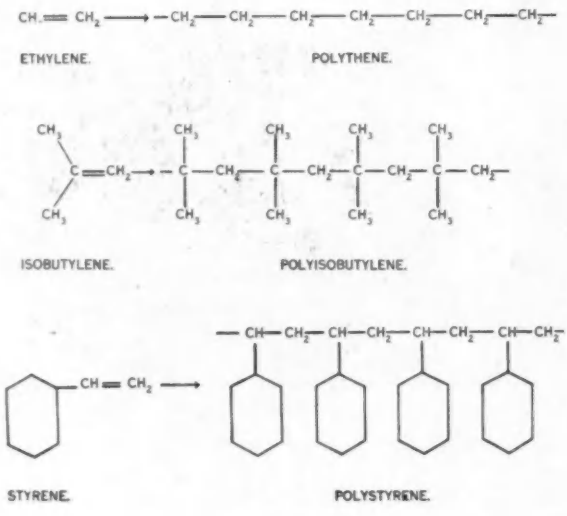


Fig. 12.—Hydrocarbon plastics.

materials; vinyl chloride can be obtained by direct union. The acetate can be similarly prepared and it is interesting to note that the properties of the two polymers are to some extent complementary and that a

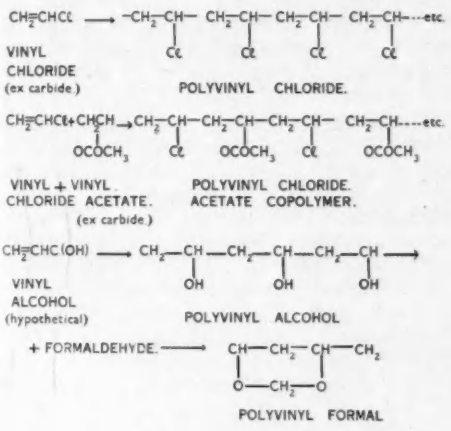


Fig. 13.—Composition of ethenoid resins.

co-polymer of the acetate and the chloride is available having some of the virtues of both. The polymers may be built up from a variable number of molecules, perhaps 75—250; the lower the polymer the softer and more rubber-like it is but, although rubber-like and used as a rubber substitute, the material is not a synthetic rubber for it does not resemble rubber chemically as do the real synthetic rubbers. In their turn, these are in a sense Plastics, but they are now usually called "elastomers." These materials were fully discussed in a previous article.*

Polyvinyl formal, shown also in fig. 13, is produced by the interaction of polyvinyl acetate and formaldehyde and has been used extensively in the U.S.A. as the coating on "enamelled" wire for electrical purposes.

* "Substitution among materials used for Cable Insulation," by Dr. Harry Barron. *G.E.C. Journal*, Vol. XII, No. 2, August, 1942, p. 71.

Starting again with the vinyl group and adding another chlorine atom thus: $CH_2=C(Cl)Cl$ gives the compound known as vinylidene chloride the polymer of which, polyvinylidene chloride, has great possibilities as a constructional material for non-metallic tubing and is being used under the name "Saran" in the U.S.A. for this purpose.

Acrylic Esters.

Another very important class of plastic is that known as the acrylic esters. The best known of these is the I.C.I. product "Perspex," obtainable as sheet or rod and as "Diakon," a moulding powder. It can be made by the series of steps illustrated in fig. 14. Ethylene, $CH_2=CH_2$, is first allowed to react with hypochlorous acid, $HOCl$, to form ethylene chlorhydrin. Hydrochloric acid is eliminated to give ethylene oxide and this is treated with hydrocyanic acid, HCN, to give ethylene cyanohydrin. Water is eliminated and the resulting acrylic nitrile is hydrolysed to produce acrylic acid. The methyl (CH_3) ester of the acid is then formed and this may be further methylated to give methyl methacrylate. The polymer of this substance, polymethyl methacrylate, is known commercially as "Perspex" and its uses range from the nose of a bomber to the lens of a cheap camera. In fact, its optical properties have enabled it to displace glass in a number of ways but its

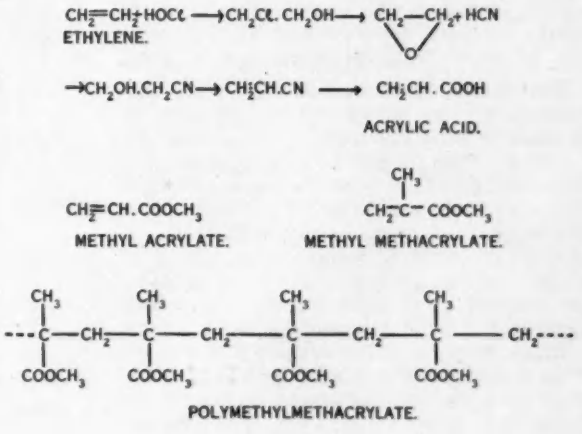


Fig. 14.—Synthesis of acrylic resin.

poor surface hardness is of course a drawback. For its use as a lens a technique of applying a thin film of glass or quartz to the surface has been worked out. Promising developments in its use for the manufacture of artificial dentures have also taken place.

Detailed mention of a number of other plastics must of necessity be omitted in a short article nor can a full account be given of the many varied applications of those plastics to which reference has been made, for example in the paint industry and as new materials for impregnation and glueing wood, fabric and other laminated materials.

Polyamides.

Nylon.

In an article concerned essentially with the chemistry of plastics it would be impossible to omit some special reference to what is one of the most amazing triumphs of the chemist. In 1929, W. H. Carothers and his colleagues of the Du Pont research laboratories began the study of high polymeric substances. Nylon, first synthesised in 1935, was the direct outcome of this work. The name is a generic one covering the group known as the super-polyamides. An amide is an organic compound containing an NH_2 grouping, a polyamide is a polymer of such compounds, while the term superpolyamide was reserved by Carothers for such materials having a molecular weight of 10,000 or more.

Turning to organic and biological structure, Carothers reasoned by analogy with such materials as horns, finger nails, silk, wool, etc., that a strong flexible plastic should result from a synthetic compound having a protein-like structure polymerised to give a chain.

The proteins form one of the major components of living matter and, though they are themselves fairly complex, they are built up essentially from simpler compounds known as amino-acids. This term might be thought to contain a contradiction of ideas for it is almost as if one were to say alkali-acid. In fact, the molecule of amino-acid is really acid at one end and alkaline at the other and like bar magnets, the molecules may be joined together, like joining to unlike. In fig. 15 the formulae of some of the simpler proteins are given and it will be noticed how orderly is the progress in passing from one member of the group to the next. In 1907 Emil Fischer produced from the simple amino-acids large molecules which he called polypeptides having many of the properties of the proteins, the linkage between each chain being the amide group, $-C(=O)-NH-$. A molecule composed of 15 glycine and 3 leucine groups was in fact prepared. Carothers probably had this in mind when he used this amide linkage to form the super-polyamides, linking up carbon chains containing 5 to 7 carbon atoms, and consisting of as many as 70 of the short carbon chain constituents.

The substances chosen as starting materials for the industrial production of Nylon were two comparatively rare chemicals, adipic acid and hexamethylenediamine. The technical production of these was worked out from one parent substance, benzene, and the steps involved are shown in Fig. 16. The working out of methods for the large scale production of these materials was a major part of the whole research and the next step was in bringing about the proper conditions for the condensation of the two substances so as to control the molecular weight and the viscosity of the final product. The first stage is the union of two molecules with the elimination of water, as shown in i, fig. 17. The terminal groups of the newly formed compounds are, however, again an amino and an acid grouping, so the whole operation can be repeated as shown in ii, fig. 17.

The molecule is now rapidly increasing in size and by a continuation of this process it is possible to build up molecules having a molecular weight of 7,000—21,000, and so obtain a super-polyamide. It is, of course, essential to keep the molecule linear for there

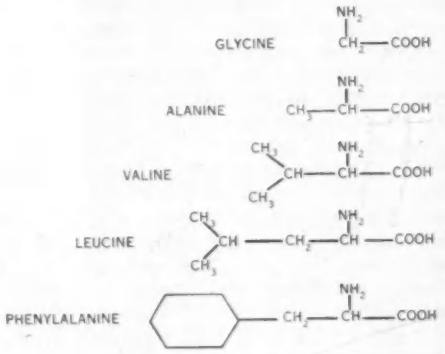


Fig. 15.—Structural units of the proteins.

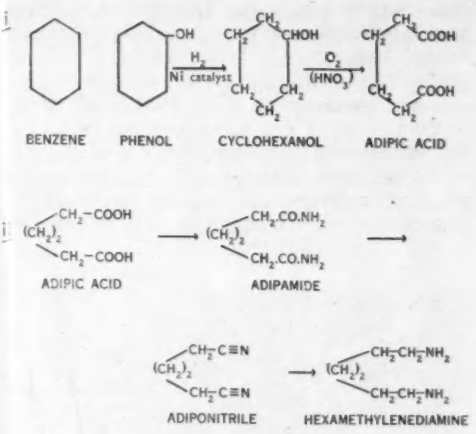


Fig. 16.—Starting materials for nylon synthesis.

is a danger of its becoming a cyclic substance as shown in iv, fig. 17. This difficulty was surmounted too; it was found that the formation of a cyclic compound could be prevented if the number of CH_2 groups in the diamine was equal to or greater than 4 and those in the dicarboxylic acid not less than 3. For successful polymerisation $m + n$ should be not less than 8 and preferably 9. The typical polyamide chain of Nylon is shown in iii, fig. 17 and the plastic is a polyhexamethylene-adipamide built up of recurring units of this type.

The synthesis is an outstanding example of a structure built to give exactly the desired physical properties and will rank as a classical piece of work. Nylon is the first plastic whose properties were in general planned or conceived before it had been brought into existence and it may be the forerunner of others.

At present Nylon is supplied mainly in fibre or filament form and the method of spinning is not from solution but from the melted mass. The molecules in the thread are arranged in a random manner, but by cold-drawing the thread orientation of the molecules is brought about and the maximum tensile strength is developed. This effect is shown very clearly in the two X-ray diffraction photographs in fig. 18. In (*a*) the narrow arcs and spots constituting the X-ray diagram show that a high proportion of the molecules are arranged in a regular manner along the fibre axis. In (*b*) the continuity of the rings shows that the arrangement is completely random.

Fibres up to 1/16in. are available at present, and Nylon promises to play an important part in the textile industry; the fibres combine a high degree of elasticity with a tensile strength greater than that of any of the usual textile fibres. In addition to this it has the appearance and lustre of silk and is more uniform. Its wet strength is equivalent to the dry strength of natural silk and it will stand a greater elongation. It is very stable chemically and shows great resistance to washing and cleaning agents.

There is a very interesting link with biological material which it may not be inappropriate to mention at this point. The two X-ray photographs of Nylon showed disorder and a high degree of order respectively before and after cold drawing. Fig. 19 shows X-ray patterns of human hair in the natural form and after stretching. The phenomenon is

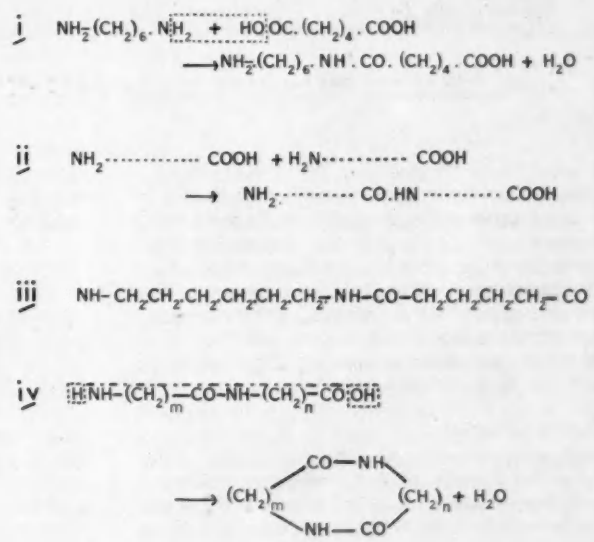
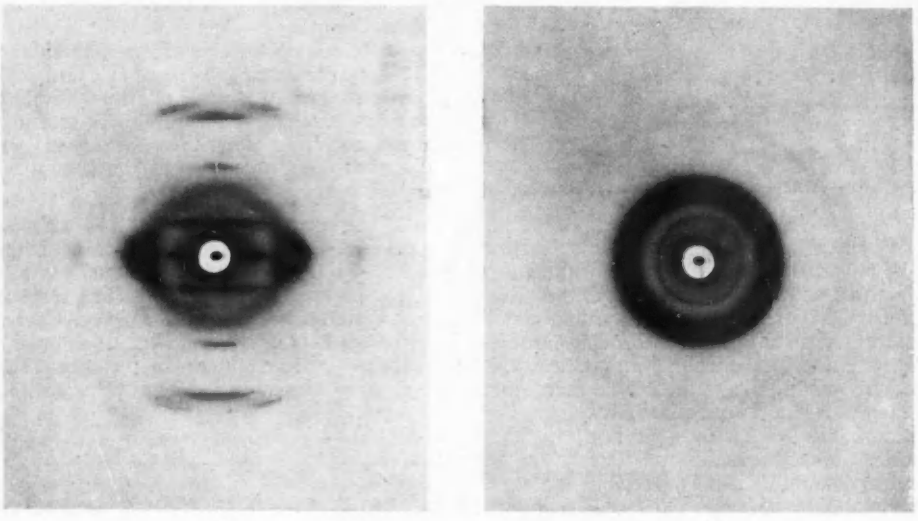


Fig. 17.—Synthesis of nylon.

somewhat similar to the drawing of Nylon, the stretched hair has some of the molecules of keratin of which it is composed regularly arranged as in the cold drawn Nylon. In "permanent waving" the hair is first of all stretched in order to orientate the keratin molecules more fully and heat is then applied, by steam or other methods, to fix the hair in the oriented and, in this instance, the waved condition. Wavy hair may be fixed in a straightened condition by a similar process. The first hosiery made from Nylon showed excessive wrinkling during the dyeing operation. This trouble was eventually overcome by subjecting the stockings to a steam treatment on a mould before dyeing; this set the stitch and no wrinkling subsequently occurred during the dyeing operation. It is possible that there is some analogy with the straightening of wavy hair!



(a) (b)

Fig. 18.—X-ray diffraction patterns (Cu Kα radiation, 3.5 cms. distance) of nylon: (a) toothbrush bristle: oriented structure. (b) toothbrush bristle heated to just above melting point: disoriented structure.

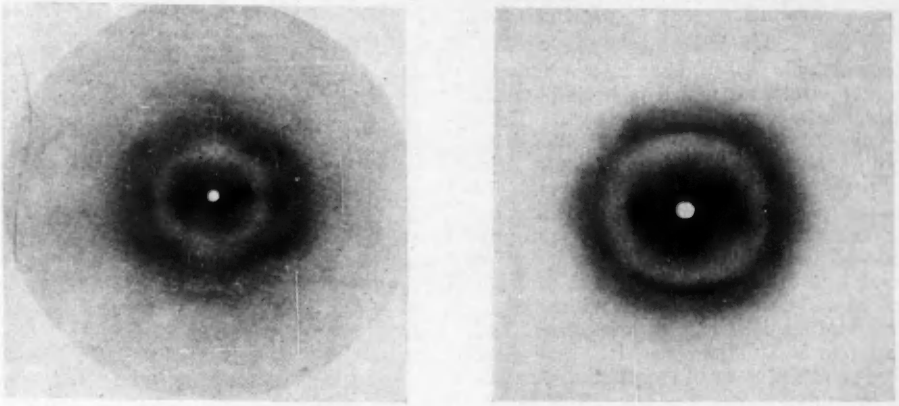
PLASTICISERS.

It was mentioned in connection with cellulose nitrate that, to produce celluloid, camphor was added as a "plasticiser" to the extent of 35 per cent or more. Similarly, polyvinyl chloride is a hard substance which has to be plasticised with perhaps as much as 60 per cent of di-butyl phthalate or other plasticiser to obtain the properties which one normally attributes to it, for example, as a rubber substitute. Some plastics are not plasticised at all and others require much smaller amounts to obtain the particular properties required, according to their use. Sometimes the formation of a co-polymer, as with polyvinyl chloride-polyvinyl acetate or with polyethylene-polyisobutylene, will go part of the way towards achieving the desired effect. But, when large amounts of plasticiser are required, as with P.V.C., it is pertinent to ask to what are the properties of the plastics to be attributed: to the P.V.C. or to the plasticiser?

In the case of the fibrous high polymers, there are obviously important reactions taking place in the "amorphous" regions of the chain which, although they are not revealed in the X-ray diffraction pattern, are yet profoundly influencing the physical properties of the polymer. A simple example may be noted in the case of the cellulose fibres. On wetting, these show no change in the X-ray diffraction pattern but the tensile strength is very considerably lowered and it is quite clear that some modification of the structure has occurred. The main changes may have taken place in the "unorganised" part of the

structure but there is at present no means of confirming this. It is likely that in the fibrous polymers, for example, there is a continuous gradation between the various parts of the structure, between highly organised and unorganised parts.

From the structural viewpoint there is still much to learn concerning the action of added plasticisers to the synthetic plastics, and in the natural materials concerning what takes place at the "unorganised" links in the polymeric chain.



(a) (b)

Fig. 19.—X-ray photograph of human hair (keratin) : (a) stretched (β-keratin); (b) unstretched (α-keratin).
(Reproduced from "Textile Fibres under the X-rays" by W. T. Astbury).

RAW MATERIALS.

Some ideas will have emerged by now as to the nature and availability of the raw materials for some of the commoner plastics, but it will be as well to consider the matter in a little more detail. A proper appreciation of the raw material situation would go far to check much irresponsible talk about plastics. The future of plastics in this country, and, to some extent, that of a wider section of chemical industry, depends primarily on the proper utilisation of our natural raw materials. The cellulose plastics, for example, either natural or chemically modified cellulose, are made from cotton linters of which an adequate supply can be had in the Empire or from the U.S.A. or Egypt. Refined wood pulp can be used and there is obviously no scarcity of cellulose in the world although some types may be more suitable than others as starting materials for plastics.

The phenols, cresols and xylenols, the basis of the Bakelite type of plastic, are obtained in large quantity from the lower boiling fractions of coal tar distillates, and a further quantity of phenol can be prepared from the benzol which is another by-product of coal tar distillation. The yield of coke oven tar from coal is of the order of 10 gallons per ton and about 3½ gallons of benzol are obtained in addition. Only about 1/3 lb. of pure phenol is obtained at present for each ton of coal carbonised and each ton of phenolic resin takes about 1,000 lbs. phenol. The demand greatly exceeds the supply and it is necessary to synthesise further quantities of phenol from benzol. Much of the phenol is required for other purposes and even the amount available for plastics does not go in its entirety to the production of the P.F. resins ; Nylon, for example, uses phenol as a starting material. J. G. Bennett in his recent lecture, "Coal and the Chemical Industry," calculates "that to replace 5 per cent of the steel and 10 per cent of the timber used in Great Britain by synthetic phenolic resins derived from coal would involve the impossible task of carbonising 5,000 million tons of coal a year, or nearly four times the total world production." This statement is, of course, based on existing methods of treating coal but it will serve to temper extravagant views on the "Plastics Age." It is possible, indeed it is imperative,

that our coal should be made to yield much larger amounts of valuable chemicals. We cannot afford indefinitely to put so much of our national wealth into the air as smoke.

Most of the other plastics depend, in this country at any rate, on coal in one form or another. Indeed we have no other large scale source of carbon; the U.S.A. has both petroleum and natural gas. A plea is being made at present in some quarters for the importation of crude rather than refined petroleum so that we may reap the benefit of the by-products obtained during the refining processes, and from which the U.S.A. obtains a valuable source of important chemicals.

Formaldehyde is produced synthetically from methyl alcohol which is itself obtained from carbon monoxide and hydrogen. These are obtained from "water gas" and the methyl alcohol is obtained by heating these gases under pressure in presence of a catalyst. The reactions are shown in fig. 20. In the same figure are set out the reactions for the production of urea from ammonia and carbon di-oxide, and of acetylene from carbide made from lime and coke. It will be obvious that so fundamental a combination of carbon and hydrogen as is found in acetylene is a good starting point for many of the plastics, particularly the vinyl series which, as was shown earlier, is based on the grouping $CH_2{=}CH{-}$. This group has been included at the bottom of the figure for purposes of comparison.

The absence of a cheap supply of carbide in this country may have hindered the development of the hydrocarbon plastics. A new interest in hydro-electric schemes designed to utilise the water resources of Scotland may eventually lead to carbide production on a large scale. On the other hand, doubts have been expressed as to our ability to compete with the resources of Canada in this matter and it may be that some of our chemical industry will have to be thought of in terms of the Empire rather than of this country alone. Much depends on our intelligent use of coal. Those countries in which mineral oil is found in quantity, especially the U.S.A., have a rich source of carbon, the primary raw material of the plastics industry, both in the oil and in natural gas, for the gas is very rich in hydrocarbons from which, by "cracking," various unsaturated compounds such as ethylene, propylene, etc. may be obtained. Butadiene, one of the fundamental compounds in the synthetic rubber production is obtained by this means in the U.S.A. and it is obvious why developments in that country have been so considerable. The "elastomers" or synthetic rubbers are related in composition to natural rubber and are not usually classed with the plastics, but it is interesting to point the relationship and to emphasise the extent of the field now covered by these modern synthetic materials, and, perhaps most important of all, to demonstrate the common starting point or raw material from which they are all derived.

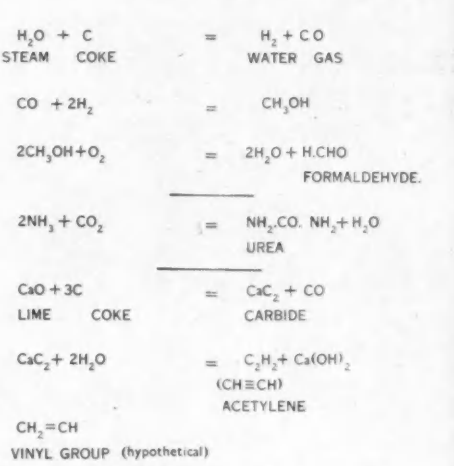
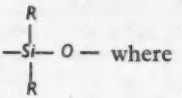


Fig. 20.—Primary raw materials.

FUTURE TRENDS.

Finally, it may be of interest to consider for a moment a new type of plastic that has been developed very recently, for it may be significant, chemically, of future trends. The weakness of all purely organic compounds is that their heat resistance is poor. In ordinary language they char or burn at quite low temperatures. Few organic materials will stand a temperature greater than 150°—200°C. for any length of time. Inorganic materials such as quartz (silicon dioxide) are unchanged in composition on heating to temperatures at which they will fuse or become plastic. It happens that silicon has the property of forming chain molecules too. The analogy cannot be taken too far for inorganic thermo-plastics cannot be said to be chain molecular in structure, but it is a fact that silicon in particular has an equal combining power with

carbon, it is quadrivalent, and it has been found possible to introduce it into an organic molecule. Silicon tetrachloride can be made to react with methyl magnesium chloride by the well known Grignard reaction to give dimethyl silicon chloride and this by hydrolysis to the silicol, dimethyl silicon hydroxide. By condensing molecules of this compound as shown in fig. 21 a chain may be built up and finally the beginnings of a network polymer with silicon atoms constituting the backbone of the molecule. This at once confers much greater heat resistance on the plastic, for the important thing to notice is that these compounds have no carbon to carbon linkage, these have all been replaced by the element silicon. The structural unit of the Silicones, the name which has been given to this class of compound, is pictured as  where R stands for some organic radical such as CH_3. These compounds have been developed in the U.S.A., and they are said to withstand a temperature of 200°C. for several days without change, and in vacuum they can be raised to 550°C. without any change other than a slight discoloration. They are highly thermo-setting and have very good electrical properties.

This latest type of organo-metallic compound promises to be very interesting, especially to the electrical industry, and is but one more sign of the breaking down of those artificial barriers which man has set to knowledge, in this instance, between organic and inorganic chemistry. Already boron, too, has been made to react with macro-molecular substances such as the cellulose compounds and with some of the polyvinyl compounds containing hydroxyl groups. The products so formed are infusible, insoluble boric acid compounds which may be made to react with water to regenerate the original thermoplastic resin.

It is hoped that this survey of the chemistry of plastics will have made clear the nature and origin of many of the new materials, and some of the old, of which so much is heard to-day. They have not been mentioned in the order in which they were discovered nor have all those that are known or are in use been included; the order chosen was that in which it seemed the chemistry could most logically be explained. Very little has been said about the properties of the various plastics but the information is outside the scope of this article and is already available elsewhere. The purpose was rather to give a clearer picture of the chemical simplicity and beauty and, above all, of the

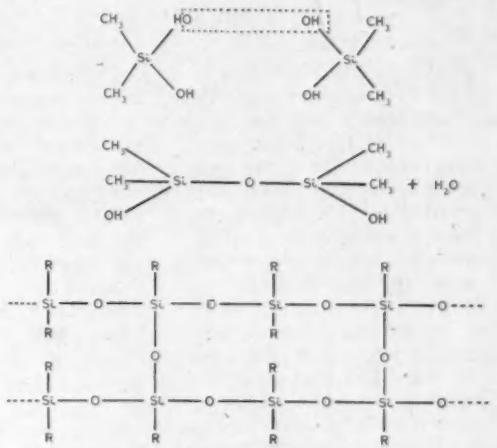


Fig. 21.—"Silicone" resins.

inter-relatedness of many of the plastics, not only to each other but also in some instances to those materials of which man is so gloriously made.

ACKNOWLEDGMENTS.

For much of the information contained in the article the author has drawn freely from the following publications:

"Plastics." V. E. Yarsley and E. G. Couzens. Pelican Books.

"Plastics, Scientific and Technological." H. R. Fleck.

"Proceedings of the Plastics Group." Vol. III, 1942–3. Society of Chemical Industry.

"Plastics for the Engineer." W. A. Kirkwood and P. D. Ritchie. (A. Reyrolle & Co., Ltd.).

"Coal and Chemical Industry." J. G. Bennett. "Reavell Lecture," Institution of Chemical Engineers, 1944.

Magnetic Dust Cores.

By G. R. POLGREEN, B.Sc., M.I.E.E.,
Salford Electrical Instruments, Ltd. (G.E.C.)

Following a general discussion of the magnetic properties obtainable by various degrees of subdivision of core material (e.g. into laminations, wire or particles), the author reviews the various methods employed for the manufacture of dust cores and gives examples of the uses to which such cores may be put. This article is reprinted from The Post Office Electrical Engineers' Journal, Vol. 37, Part 1, April, 1944.

INTRODUCTION.

IN the early days of electrical engineering, the necessity for the subdivision of magnetic cores carrying alternating magnetic flux was soon apparent from practical considerations alone. Even at a frequency of 50 c/s, the eddy current losses induced by the flux in the magnetic core, acting as an electrical conductor, were sufficient to cause considerable heat and loss of efficiency; but there was a simple remedy, the building up of the magnetic cores in the form of stampings insulated from each other in one direction at right angles to the flux. It was also found that eddy current losses could be reduced by increasing the electrical resistance of the magnetic metal from which the stampings were made and these were for many years the only two practical methods of realising the possibilities of magnetic cores for the numerous potential uses in the telephone and radio frequency ranges.

During recent years, however, the technique of making magnetic cores from fine magnetic powder or dust has completely altered the position, and the numerous advantages of ferro-magnetic cores are now being extensively applied for a great variety of purposes up to the higher radio frequencies, using the basic principles that are commonplace for electrical machine and equipment design at power frequencies. The considerable time taken in achieving this progress is due more to the technical and engineering problems involved, than lack of appreciation of requirements or to the assumption that air was the only suitable medium for magnetic flux generated by frequencies in the telecommunications range. Thus, over 60 years ago, Heaviside proposed a magnetic core made from iron filings set in wax, but with present knowledge such an arrangement is basically unsound, because the permeability of the "core" would be very little different from that of air.

REQUIREMENTS.

Modern dust cores are made from highest grade magnetic materials and give very low total losses at the working range of frequencies, so that the "goodness factor" or Q ($= \omega L/R$) of a dust cored inductance can be made, if properly designed, to the same order of value from 50 c/s to 50 Mc/s. To cover this wide frequency range a number of core types are required, and a number of permeability grades of core material, the intrinsic permeability values varying from 12 for the highest frequency, and 120 for the lowest frequency.

But for all these numerous requirements, the principles underlying the construction of the dust cores are the same, and may be summarised into three separate stages of manufacture:—

(1) The magnetic metal or alloy is produced in a finely powdered form with suitable size, magnetic, electrical and physical properties.

(2) This powder is insulated by coating the particles with a tenacious layer of insulating material.

(3) The insulated powder is pressed at high pressures into moulds under conditions which retain the insulation properties of the coated particles even though considerable distortion of particle shape may take place.

The finished product must conform to a number of practical requirements which are universal in the telecommunication field :—

(*a*) Adequate mechanical strength to withstand handling in coil winding departments and test rooms.

(*b*) Stability under varying conditions of temperature, atmosphere, and for indefinitely long periods of time.

(*c*) Constancy of electrical properties under extreme conditions of electric or magnetic fields.

The satisfactory solution of these problems has been the result of very extensive research and development work in many directions ; chemistry, metallurgy (in its general and specialised forms of powder metallurgy), microscopy, as well as magnetism and the widespread technology of electrical testing. The extent of the problem is evident from the wide range of patent literature covering all the above aspects of the subject.

METHODS OF CORE DIVISION.

Before considering the properties of modern magnetic dust cores, it is useful to consider critically the various methods of subdividing a solid magnetic core and the effect on the magnetic and electric circuits. For this analysis consider ring-shaped specimens (commonly known as "toroids"), such as are used for standardising purposes, giving a symmetrical magnetic field without appreciable leakage flux. There are three main planes of subdivision (fig. 1) which can conveniently be termed radial, concentric and transverse, the first being at right angles to, and the second and third parallel in two different planes to the direction of the magnetic flux. Either transverse or concentric sub-division has the effect of greatly reducing the eddy current losses by insulating the individual laminations with an insulating layer which, if suitably thin, will not appreciably reduce the flux-carrying capacity of the core. In other words, the permeability and magnetic characteristics are practically the same under low frequency A.C. conditions as would apply to direct current testing. Transverse laminations are commonplace for all 50 c/s machinery and equipment, and need no further comment. The concentric type can only be achieved in practice by winding a spiral of insulated magnetic tape, which results in the introduction of a continuous non-magnetic gap, but of low reluctance, into the magnetic path, reducing the effective permeability of the core but increasing the magnetic stability without distorting the flux. The next step in subdivision is the combination of spiral and transverse methods which results in a spiral of insulated magnetic wire ; this results in a further decrease in effective permeability and appreciable decrease in the space factor of magnetic material, hence a fairly stable but rather inefficient magnetic circuit. Finally,

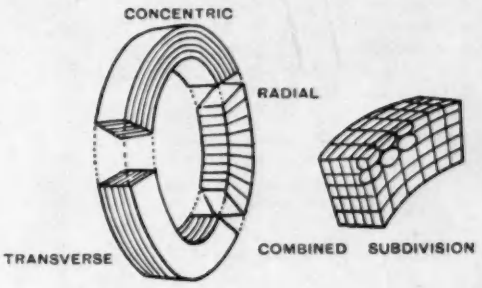


Fig. 1.—Methods of sub-dividing magnetic cores.

if the magnetic wire is chopped up into small pieces by the addition of a radial method of subdivision, the permeability is still further reduced, but the magnetic stability is thereby greatly improved.

The dust core is the practical version of the last combination of the three methods of subdivision and the magnetic characteristics can therefore be deduced from these considerations, together with the inherent properties of the magnetic metal itself.

To appreciate the extent of the subdivision it is necessary to realise that the eddy current loss is proportional to the square of the frequency. The present applications of cores for telephony cover one thousand times, and for radio one million times the normal power frequency of 50 c/s, so the extent of comparable subdivision must be 10^{-6} and 10^{-12} respectively for telephone and radio practice in comparison, for example, with ordinary 16 mil silicon iron stampings used for mains transformers. This shows clearly why very few of the alternative methods of subdivision, spiral strip or wire, can be important in telecommunications applications. To indicate the extent to which this problem of subdivision has been solved, a typical plug core, as used in vast quantities in radio equipment, and with dimensions $\frac{3}{4}$in. diameter by $\frac{1}{4}$in. long, contains about one thousand million separate magnetic particles, each of which must be

properly insulated from its neighbours, in order that the eddy current loss shall be limited to a reasonably low figure.

MAGNETIC PROPERTIES.

The reduction in permeability of the magnetic core by the introduction of innumerable small air gaps results in a very high magnetic stability, as will be seen from the diagram (fig. 2), and Table I summarises the various advantages and disadvantages of stampings, air and dust cores, in so far as the magnetic properties are concerned, and apart from the reduction of eddy currents just described.

The following points are of great importance :—

(1) The dust core permeability curves are close approximations to straight lines, especially for lower permeability types, and in this respect they behave as air cores. This applies even at saturation values of H, which are, however, so great that the heat generated is in practice a limiting factor.

(2) The hysteresis loss is of the same order as that of the solid metal, but the shape will be that of an extremely elongated ellipse. Hence, the incremental permeability, or the permeability due to the simultaneous effect of A.C. and D.C. fluxes will be constant at approximately the same value as the core permeability. This is a vast improvement on all forms of stamping cores.

(3) By altering the "effective air-gap" of the dust core, *i.e.*, by altering the amount of insulating and filling materials, the core permeability can be varied over a fair range with the same magnetic metal ; the advantages in (1) and (2) above apply to the whole range and the cores can all be made symmetrical magnetically, *i.e.*, with virtually no leakage field.

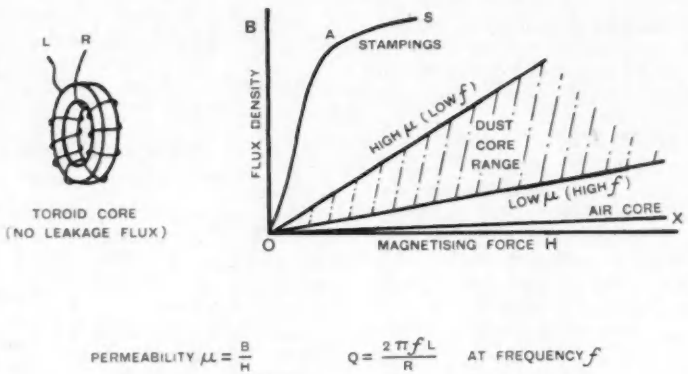


Fig. 2.—Core Types—Magnetic Properties.

OAS represents a typical magnetising curve for a solid magnetic core. It is generally known that the permeability B/H varies considerably over every part of the curve and is indicated by the slope of the curve at any particular value of magnetising force H. OX is the magnetising curve of an air gap, that is to say, the permeability is constant at unity, and the hysteresis loss is zero. The shaded area represents the present range of dust cores, being the resultant of the curves for the solid metal and for the air gap, the actual permeability being dependent on the ratio of magnetic to non-magnetic material in the core. This can be varied over a wide range by using coarse powder and very little insulating material for high permeability cores, and fine powder with non-magnetic fillers, in addition to the insulating material, for low permeability cores.

In spite of the predominating role of the "effective air-gap" in deciding the permeability of the dust core, the intrinsic permeability of the magnetic metal is of considerable

importance for high permeability cores when the thickness of insulation may be as small as a fraction of a micron (·001 mm).

MATERIALS AVAILABLE.

So far only the effect of subdivision of the dust core has been considered; now the available magnetic metals and alloys available will be examined. The desirable properties of the solid metal for dust cores are :—

(*a*) High permeability.
(*b*) Low hysteresis loss, especially for cores in telephone circuits and where high flux density occurs.
(*c*) High specific resistance, which limits eddy current and enables lower degree of subdivision to be utilised. This is of special importance at radio frequencies where hysteresis loss is normally of small importance.

Iron is, of course, the most common and universal magnetic metal, and this has been widely used for all types of dust core. The disadvantage of iron is that the permeability and the specific resistance are low and the hysteresis loss is high in comparison with modern alloys. Very pure and special types of iron, such as is made from iron carbonyl, give very low hysteresis losses and are available in very fine powder, which means that there is a considerable use for this type of material. The well known silicon iron alloys have been made in quantity in powder and dust cores, but their advantages over iron are small for these purposes, and the disadvantages in comparison with high grade alloy cores are very numerous.

There are very many different alloys which give remarkable magnetic properties, but for this analysis it is convenient to divide them into two groups, both widely used under a variety of names in sheet and stamping form: "high nickel alloys" containing over three-quarters nickel, and the remainder mostly iron; and the "low nickel alloys" containing approximately equal quantities of nickel and iron. Both groups frequently have additions of other metals, which give, or are claimed to impart, various advantages in regard to both hysteresis loss and resistivity, but the general properties of each group, with or without the additions, can be compared advantageously.

The "high nickel" group, in the form of stampings, gives permeability values between 10,000 and 100,000, many times greater than that of silicon iron; the "low nickel" group gives much higher permeability in comparison with the iron group, but not so great as the high nickel alloys. Alloys of the second group have specific resistance values nearly 10 times that of iron and they are more economical of the comparatively expensive metal nickel. All the alloys in both groups are rustless, which is a considerable practical advantage. The disadvantage of all these nickel-iron series of alloys is that they are very sensitive to mechanical strain and must be subjected to critical heat treatments to yield their advantages, but methods of manufacturing powder and cores from these groups of alloy have now enabled the full advantages to be obtained.

TABLE I

Type	Advantages	Disadvantages
Stampings.	High permeability. Low copper loss.	μ varies continuously from 0 to saturation. Losses limited only at low *f*. Magnetically unstable saturates at medium H.
Air Core.	No hysteresis loss. No saturation.	Excessive copper loss. Unwieldy size for working Q values. High temperature coefficient.
Dust Core.	Controlled μ and losses over wide *f*. Constant μ for A.C. and combined D.C. High magnetic stability.	Upper limits to μ for adequate *Q*.

CORE MANUFACTURE.

In the construction of the dust core, the first consideration is the magnetic dust itself. All the magnetic materials briefly reviewed have the property of toughness rather than brittleness, and this factor greatly complicates their production in the form of fine powder. It may be thought that modern grinding machinery will mechanically reduce any metal in ingot or pellet form to fine powder, but this is not the case, unless the metal is melted together with an embrittling agent. However, there are a number of other methods which will enable metal or alloy powders to be made. Special machines have been developed which disintegrate such metals by melting them

and blowing them in molten state into cooling liquids. Other processes operate by drawing the materials into fine wire and then chopping up, subsequently projecting streams of the semi-disintegrated metal against each other or against metal plates. Chemical methods of reduction of pure compounds of the metals are widely used for producing a variety of magnetic powders and these yield great advantages in purity and fineness of the resulting product. A further process to chemical reduction of oxides is the production of the metal carbonyl in liquid form, which is subsequently volatilised and deposited in the form of tiny metallic spheres. The continuous electrolysis of metal compounds in solution can be adapted to yield fine spongy material that can be broken up by a milling process into fine powder. The last process was formerly used in the manufacture of iron dust cores for loading coils in America.

The problem of making magnetic powder is by no means limited to maximum size of particles. The shape, density, size distribution, physical and mechanical properties, must all be controlled to within close limits, and heat treatments are complicated by the fact that all metal powders tend to stick or sinter together even at temperatures far below the melting point. Considerable use is made of this phenomenon in the important new process of making metal parts out of powder, known as "powder metallurgy."

Before pressing the magnetic powder into dust cores, all the particles must be insulated with a tenacious coating which will withstand subsequent pressing and heat treatment processes. Compared with the Plastics Industry technique, the pressures are very high, being up to 20 tons/sq. in. for low permeability cores and up to 100 tons/sq. in. for the higher permeability types. The latter pressure intensity is well over the elastic limit of the metal, and the particles will therefore interlock into a solid mass without any binder whatever. The strains imposed in nickel iron cores are very considerable and they must be overcome by a high temperature annealing of the cores after pressing. The insulation film between the particles must be such that it does not break down either in the shearing action imposed during the pressing of the powder, or in the heat treatment of the core, and this presents one of the greatest problems in dust core manufacture and a limiting factor to the development of cores with still higher permeability values.

The finished cores are tested for permeability, losses, dimensions, mechanical strength and to ensure that toroids are magnetically symmetrical, and that there is no trace of external field. It is possible to keep dimensions to limits and a general tolerance for small cores is 5 thousandths/inch, by careful design of press tools and the close control of the insulating and finishing processes of the powder and cores. Different stages in the production of toroidal coils for loading coils and filters are illustrated in fig. 3.

APPLICATIONS.

The first practical application of dust cores on a large scale was by the Bell Telephone Laboratories in America in 1915 for the loading of underground cables. Iron powder made by an electrolytic method was insulated with shellac and pressed into thin ring cores with a permeability of 35. These cores superseded iron wire in the construction of loading coils, the wire giving insufficiently low loss at telephone frequencies and instability of inductance. Air cored toroidal cores had been used in some instances, but they were too unwieldy for most practical applications. Even the iron dust cored coils were of considerable size to give the required electrical performance, and the weight of a loading pot containing a number of these was of the order of one ton or more. Such pots are connected to the telephone cable at intervals of 2,000 yds., and thus the construction of manholes and the installation was a considerable undertaking. The work of Pupin in 1900 in specifying the requirements of the lumped inductance loading of telephone transmission lines is too well known to require a summary of it in this article, but it is sufficient to say that the inductance coils must be of such quality that they do not appreciably add to the effective resistance, capacitance or crosstalk of the circuits. Further advances in loading coil design were made by the introduction of nickel-iron alloys of the permalloy type, which resulted in cores with permeability values of over 100. This reduced the size of cores by about 70 per cent for the same losses, and gave increased stability, lower self-capacitance, and lower crosstalk, and the higher cost and complication of core production was greatly offset by these extensive advantages. Thus the development begun in 1928, in conjunction with the improvements in the telephone repeater and in cable construction, has resulted in the extension, on a considerable scale, of the use of dust core loading coils throughout the whole underground telephone cable net-

work. During the last ten years, not only have all main trunk line audio cables been loaded with these new coils, but it was found that the performance and design of toll cables, and even junctions, could be greatly improved by this means. To-day almost all audio communication in the underground telephone transmission system of the Post Office is by means of cables loaded with coils wound on alloy dust cores.

replacing the conventional variable air condenser with a fixed capacitance. About the same time Vogt in Germany developed a material known as "Ferrocart," which had very low losses at radio frequencies and this could be used for the construction of high Q coils with low leakage for all radio filters, transformers and chokes.

Ferrocart consisted of insulated iron powder coated on to bands of paper, which were then

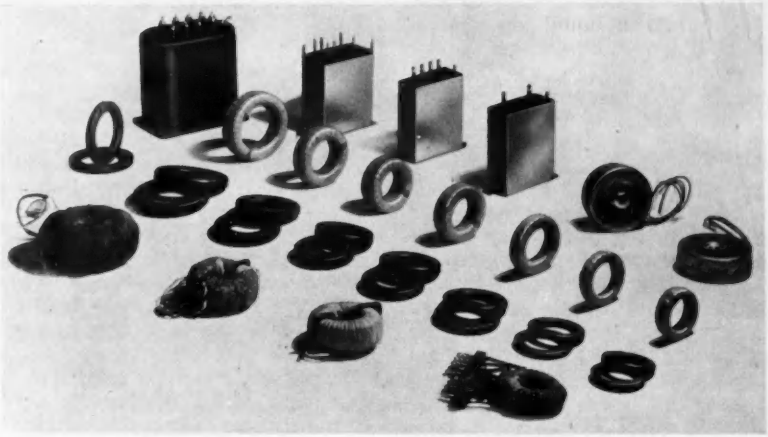


Fig. 3.—Toroidal cores and coils for loading coils and telephone filters.

In 1930, carrier telephony on underground cables was becoming a practical proposition and dust cores became an essential part of filter design. At that time it was considered that the upper limit of frequency for dust cores would be about 8 kc/s, but improvements in core design kept pace with the expansion of the carrier frequency band, from audio frequency up to 60 kc/s, with the introduction of the 12-channel system. For this, the use of coil loading was discontinued because it imposed an upper frequency limit to the transmission band and extra repeaters were used to overcome the attenuation. The same advantages which dust cores give to loading coils—low losses, zero leakage, low self-capacitance, compactness—are those required for the inductances and transformers for telephone exchange and repeater equipment.

The application of dust cores to radio equipment dates from twelve years ago, when Polydoroff published in America his work on what he called "permeability-tuning"—a method of tuning by altering the inductance of a coil by moving a core inside a solenoid and cut into fixed lengths and formed under hydraulic pressure into sheets; alternatively, the paper was wound on a spiral to make tubes and rods of magnetic material.

This material could be cut up or drilled in much the same way as ebonite, and so a large variety of shapes and sizes of magnetic core could be made up without difficulty. The intrinsic permeability (as measured by toroidal fieldless cores) was about 10, and Q values of 200 or more were attained at frequencies of 100 to 3,000 kc/s. This magnetic material showed great promise due to its performance and working properties, but the advantages of technical performance were offset by a severe limitation of stability due to the wax incorporated in the material to bind the layers of coated paper together. Ferrocart became obsolete when new methods of moulding dust cores from insulated powder in automatic machines were introduced about 1935. These cores, which are similar in properties, performance and stability to the dust cores made for loading coils and carrier coils, are made in a great variety of sizes and

shapes, and have found very extensive applications in all branches of radio equipment. Due to the lower pressures involved in the making

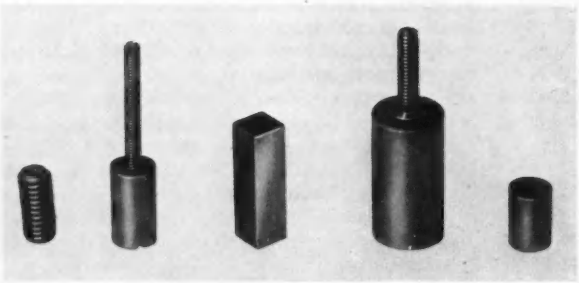


Fig. 4.—Plug cores for fixed and adjustable radio inductances.

of these small cores for radio frequencies, it is possible to manufacture them at comparatively high speeds in automatic presses, provided that the shapes are simple and regular. Composite cores can, however, readily be made by cementing several pressed pieces together.

ADVANTAGES OF DUST CORES.

The advantages compared with air-cored coils can be briefly summarised :

(1) High Q for same size.
(2) Use of adjustable inductance (screw cores, brass insert cores, etc.).
(3) Variable inductance (" permeability tuning").
(4) Self screening to considerable extent (ironclad cores).
(5) Low or controllable temperature coefficient.
(6) Suppressor cores with heavy D.C. magnetisation, and appreciable temperature rise.

Radio cores have been used up to well over 50 Mc/s. with considerable success, and for these higher frequencies the open type magnetic circuit or plug cores (fig. 4) is the type generally used. For the lower radio frequencies, however, the closed magnetic circuit core (fig. 5) has considerable advantages, especially when the coil is wound on a low loss bobbin of polystyrene.

In considering the application of dust cores for any purpose, it is necessary to study the performance of the complete coil as a self-contained electrical unit. Equipment designers sometimes complain that they cannot obtain the performance of the dust cores claimed by the manufacturers, but the usual reason is that they have overlooked the fact that the core is one of many links in a chain of materials which affect the ultimate electrical performance. Thus, the core insulating material or bobbin must be of suitable loss material, and the wire must be stranded to a suitable degree in order to ensure that the eddy current loss in the copper wire is of the same order as the core losses. The screening case must be of correct dimensions and construction, and the impregnating compound

Fig. 5.—Typical iron-clad dust cores, showing construction.

must be of suitable dielectric and physical properties. It is possible to analyse these losses by testing a coil at a range of frequencies to give the eddy current factor at several current values, to give hysteresis loss and by extrapolation to zero frequency to obtain the "magnetic viscosity," a factor of considerable importance for low frequency applications. Hence, the full details of the core performance can be assessed by the core manufacturers with reasonable accuracy, and the ultimate performance of the completed coil should therefore be predictable and controlled within close limits.

The completed and sealed dust core coil will retain its inductance value to a fraction of 1 per cent for an indefinite period irrespective of any currents, A.C. or D.C. which may be applied continuously or suddenly.

CONCLUSION.

To summarise the type of core required for any particular application for any frequency band, a diagram has been drawn up (fig. 6), in which both the frequency and permeability are plotted on log scales, and thus the relative position and importance of both stamping cores and air cores can readily be appreciated. The Q values which can be obtained will, of couse, depend on the size of core and details of the construction, so the diagram should be taken as a first approximation only. It will be seen that higher permeability toroid cores are predominant at the lower frequencies, and the open magnetic circuit cores of low permeability are most important for higher radio frequencies. At medium and low radio frequencies, closed magnetic cores other than toroids, usually referred to as "ironclad" cores, give the best results, and afford a considerable degree of screening without increase of loss, and within a small space. It is important to note, however, that there will always be more external field around an ironclad core than there would be in a similar shape and type of stamping core used at power frequencies, because in the latter the "air-flux" due to the coil without the magnetic core is generally negligible, but in all radio dust cores it is an appreciable proportion of the total flux.

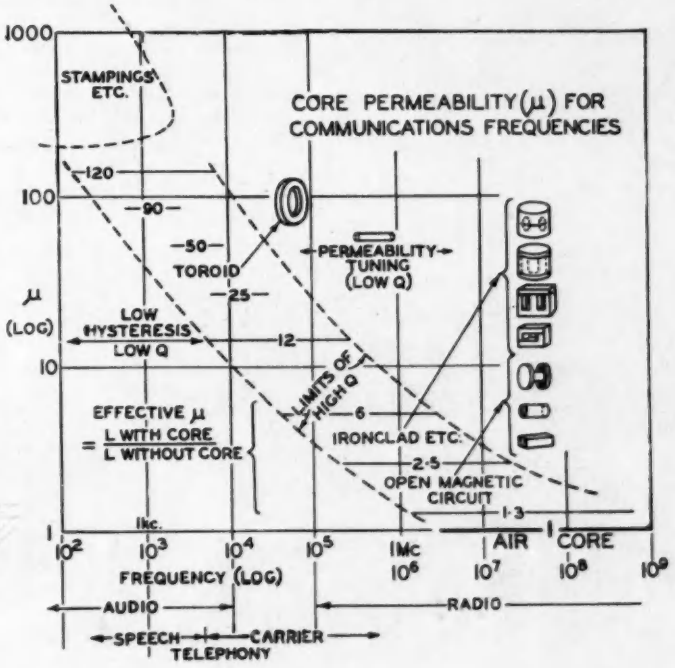


Fig. 6.—Core permeability at communication frequencies.

Normally, for medium size cores, the space between the two curves in fig. 6 denotes Q values of well over 100, which can be expected within a wide range of frequencies; for small frequency ranges the Q will be between 200 and 300, but the range can be extended and the Q increased by using larger cores and coils, especially at the lower frequencies.

For special purposes it is desirable to work outside the maximum Q range of any particular core. For instance, for very low hysteresis loss requirements, large low permeability cores are used; conversely, at radio frequencies long cylindrical cores of high permeability are sometimes used to give a wide inductance variation for "permeability tuning" In both these examples, the Q value is sacrificed in order to gain the alternative advantages, and this will apply in a variety of directions.

It will be seen from this short review of magnetic dust cores that there are considerable further possibilities in their applications to the many and varied purposes of electrical engineering.

Technical Literature.

A DIGEST OF RECENT BOOKS, ARTICLES AND PAPERS BY MEMBERS OF THE G.E.C. AND ITS ASSOCIATED COMPANIES.

MODULATION THEORY.*

By A. BLOCH (Research Laboratories).
Jour. I.E.E. Vol. 91. Part III., p. 31, March 1944.

Shows how complex notation can be adapted to the treatment of modulation problems and the advantages arising therefrom.

Amplitude, phase, and frequency-modulation appear as special cases of the most general type of modulation so that the changeover from one type to the other can be conveniently followed. The paper discusses the general problem of the passage of a modulated carrier through a filter with symmetrical or asymmetrical characteristics. A simple formula is derived (containing Carson-Fry's "Variable Frequency Circuit Theory" as a special case) which gives the resultant signal without preceding Fourier analysis if the filter characteristic is known in form of a power series. A simple and more general derivation is also given of the known method of paired echoes which is further developed into a method of unpaired echoes.

Appendix I discusses the spectrum of a frequency-modulated wave and its symmetry; Appendix II deals with the calculation of modulation products when the characteristic of the modulator is described by a trigonometrical series.

THE PLACE OF SCIENCE IN THE ART OF LIGHTING.

By R. O. ACKERLEY (Illuminating Engineering Department), and ALISTER McDONALD, F.R.I.B.A.
Trans. I.E.S., p. 45, March 1944.

An address delivered before a joint meeting of the R.I.B.A. and the I.E.S. on January 18th, 1944. The paper discusses the reasons why a knowledge of the technical aspects of lighting is essential for an architect in designing all types of buildings.

THE DESIGN OF STAMPINGS FOR LOW FREQUENCY TRANSFORMERS.

By R. MAWSON (Telephone Works).
Electronic Engineering, p. 514, May 1944.

The author derives a formula by which the relative merits of different shapes of stamping can be compared, and goes on to show how the formula is used to determine the optimum core area and position of the core, the optimum stamping when a given dimension is fixed, and the optimum shape of the stamping for a given area. A second formula determines the shape of the stamping giving maximum inductance for a given size of wire.

FUSES AND CIRCUIT BREAKERS FOR CIRCUIT CONTROL AND PROTECTION IN MARINE INSTALLATIONS.

By C. P. HARRISON (Marine Department).
Trans. Inst. Marine Engineers. Vol. LVI. Part 2, March 1944.

The importance of maintaining electrical supply to essential auxiliaries on ships has emphasised the need for adequate protection against faults or generator overloads. Such protection is afforded by circuit breakers and fuses.

Some of the latest information is here collected in convenient form regarding the working principles of different types of fuses, particularly the high rupturing capacity (H.R.C.) cartridge fuses which will become more common as a result of modified Classification and Ship Wiring Rules.

The respective functions of fuses and circuit breakers are considered together with the layout of switchboards.

* A limited number of reprints is available; copies may be obtained on application to the Editor, G.E.C. Journal, Witton Engineering Works, Birmingham, 6.